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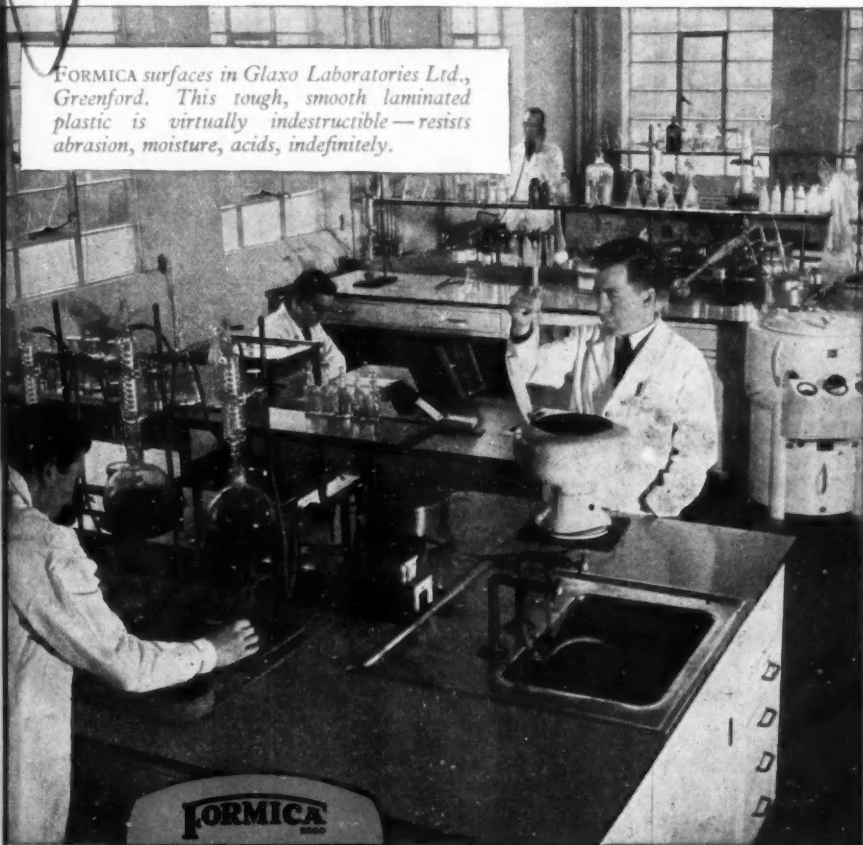
The Chemical Age

AUG 30 1952

OL LXVII

16 AUGUST 1952 DETROIT No 1727

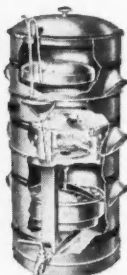
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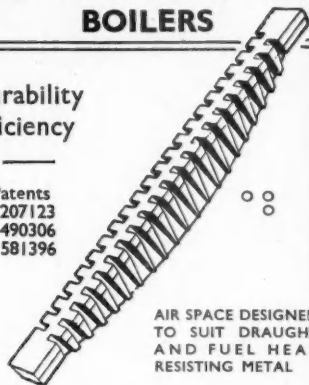
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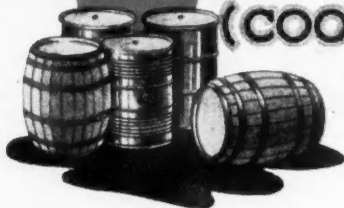


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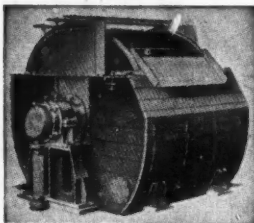
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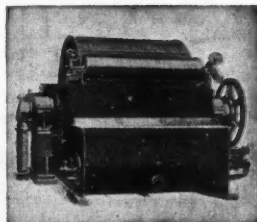
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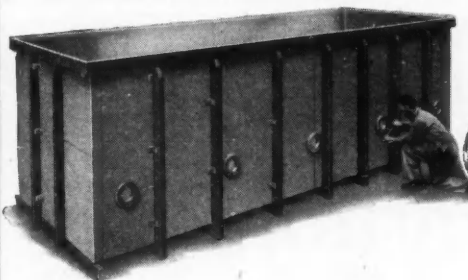


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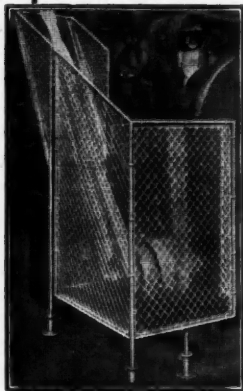
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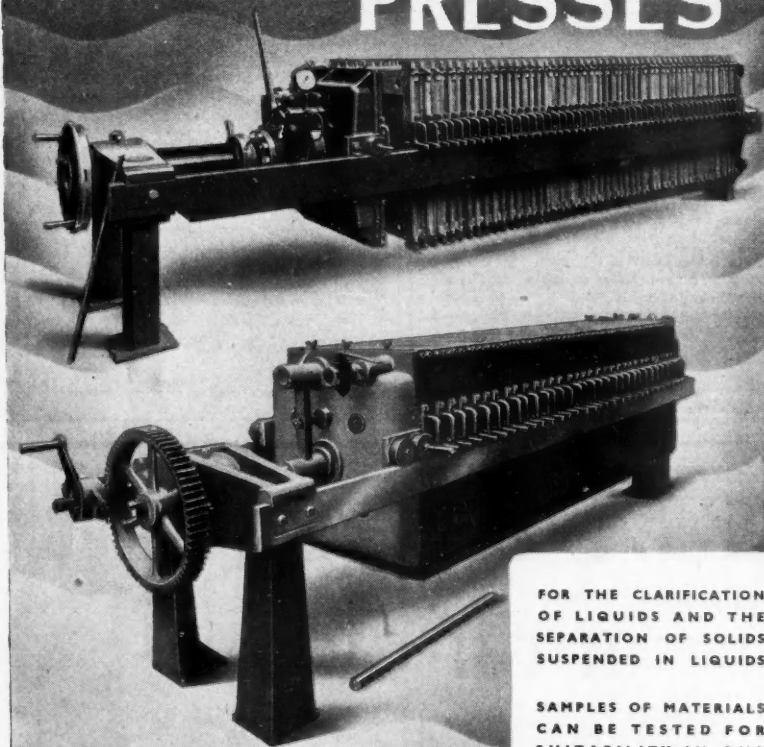
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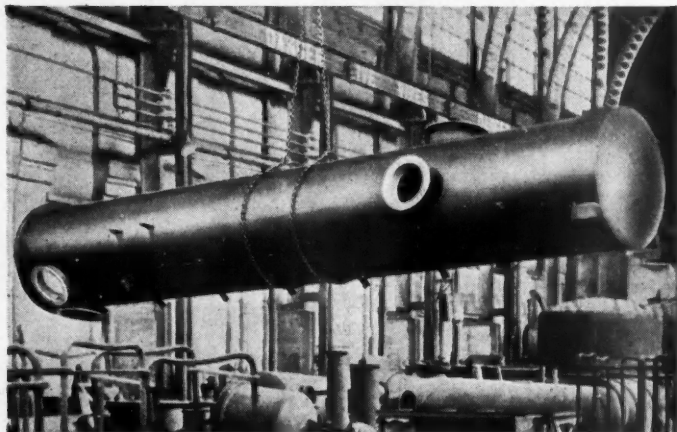
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Volume LXVII

16 August 1952

Number 1727

The Rise of Chlorophyll

THE appearance of chlorophyll in a variety of new markets will have been noticed by most chemists. Chlorophyll has suddenly come forward as a domestic deodorant, as a queller of those much advertised human embarrassments, B.O. and halitosis, and as a preventive, albeit perhaps only a mild one, of dental decay. To wonder whether these claims can be justified is not unnatural, especially when in the more readily accessible scientific journals papers about chlorophyll have not been numerous. Chlorophyll is an attractive material for commercial development. It is natural; indeed, cannot yet be synthesised. It is easily solvent-extracted from green crops and it can hardly carry any toxic hazards, for in its natural state in plants it has long been consumed by both humans and animals. Nor, as a purified extract from natural produce, is its production and utilisation new. Its employment as a colouring agent dates back to the period of World War I, if not earlier. Its medicinal possibilities date back to the 'twenties. Proprietary tonics based upon chlorophyll have long been known.

If there is in the new claims for chlorophyll a substantial measure of truth, it is clear enough that chlorophyll is a material of nature whose importance has been previously and enor-

mously under-rated. To call it a 'mystery-chemical' would not be too crude an exaggeration; after all, not much has been discovered about chlorophyll, although its significance to plant-life has long been recognised. Years of biochemical investigation have elucidated very little about this fundamental rôle of chlorophyll though without it we should have no food and no fossilised fuel. The simple nutrients required in plant metabolism are in the main those also needed for animal nutrition. To assume that chlorophyll has further services to perform after it has fulfilled its purpose in plant-growth is not an irrational speculation. But we have not yet even unravelled its molecular structure; in that research journey we are no farther than believing it to be a molecule of 55 carbon, 72 hydrogen, 5 oxygen, 4 nitrogen, and 1 magnesium atoms.

It is more than a quarter of a century since a relationship between haemoglobin and chlorophyll was first suspected. This was the initial basis of therapeutic research and from it the use of chlorophyll in treating certain kinds of anaemia was developed. There has been fairly continuous research on this particular use of chlorophyll since and the first realisation of its deodorising powers emerged in 1941 as a somewhat accidental by-product of clinical investi-

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gations. It was noticed in America that the urine of patients receiving chlorophyll was less odorous. This chance observation was followed by a variety of controlled tests. It was found that specific doses of chlorophyll reduced perspiration odours and brought about marked decreases in breath odours after onions had been eaten. At about the same time, quite separate investigations confirmed the deodorising capacity of chlorophyll. As a healing stimulant for damaged tissues chlorophyll had previously shown some usefulness; when applied to malodorous wounds during the war, it was found that the odour quickly disappeared. Since then a significant volume of evidence as to chlorophyll's deodorising properties has been accumulated.

Nevertheless, the development is not without critics. It is pointed out that the tests have been subjective in nature. But can this be otherwise in the realm of odour? We have no other way of measuring odour save by the use of that subjective instrument, the nose. How far the employment of panels of trained 'smellers' can introduce a generally acceptable standard of objectivity must inevitably cause argument. And with commercial development proceeding at a fast pace, certainly not suffering from malnutrition in its supplement of pressure advertising, a good deal of scepticism is equally inevitable. Inasmuch as controversy exists, it seems that time alone will resolve it. The public will assess the efficiency of chloro-

phyll preparations as household or personal deodorants by their own subjective experiences. Many such private tests will be hopelessly irrational, particularly in the household field. The exploration of chlorophyll's potentialities as an industrial deodorant, a field in which pressure advertising is less likely to influence subjective judgment, may produce another opportunity for critical testing.

The introduction of chlorophyll into toothpastes does not appear to rest upon anti-halitosis effects alone though controlled test evidence establishing this property have been published this year (Barail, *J. Soc. Cosmetic Chem.*, 1952, 3, 1-4). Antibacterial action is claimed for chlorophyll so far as a bacillus believed to cause caries is concerned. Also, the rate at which acid is produced in saliva-starch mixtures in the mouth is said to be reduced by chlorophyll. Here again there is likely to be controversy or argument.

Meanwhile, and particularly in the United States, chlorophyll is invading a medley of products—not only direct deodorisers but face creams, shampoos, chewing gum, indigestion tablets, dog foods and biscuits, and (for reducing throat irritation!) cigarettes. In the past three years production of chlorophyll has greatly increased—in America by more than 700 per cent. It is difficult not to feel that the devotion of one-twentieth of this effort and energy to pure research on chlorophyll would in the end yield greater benefits.

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The annual subscription to **THE CHEMICAL AGE** is 35s. Single copies, 9d.; post paid, 1s.; **SCOTTISH OFFICE:** 116 Hope Street, Glasgow (Central 3954/5). **MIDLANDS OFFICE:** Daimler House, Paradise Street, Birmingham (Midland 0784-5). **THE CHEMICAL AGE** offices are closed on Saturdays in accordance with the adoption of the five-day week by Bess Brothers Limited.

Notes & Comments

Pulp and Paper

THE recent debate in the Commons on the economic situation brought a good many 'damp squib' or 'anticlimax' criticisms; the mountains that had been rumbling beforehand produced what many people regarded as a mouse. The Chancellor did point out, however, that imports of pulp and paper would be greatly reduced in the second half of the year, akin with unrationed foodstuffs. To dismiss these restrictions as minor or meaningless is surely wide of the mark. Here, in food and in wood cellulose, we have commodities that we need but which constantly make adverse contributions to that vital balance we call the 'terms of trade.' They are both short, both being demanded in the world at a greater rate than they can be supplied. In consequence their relative price levels remain high. Even when all prices tend to fall, their prices fall more slowly and rather less. To obtain as much as we had 'the year before,' we must each year produce more of our own export articles in exchange. It is one of the fundamental causes, if not perhaps the only cause, of our recurrent economic crises that commodities we most need to buy tend to remain in a seller's market while the things we can sell tend to drift into a buyer's market. With food we at least have the chance and hope of increasing production at home; with paper and paper-making pulp there is no similar relief, however partial.

Alternative Processes Needed

IF increased food production is important everywhere in the world, so too is the development of processes that enable paper to be made from cellulose sources other than wood pulp. And if this applies to Britain in regard to food with a special urgency, so it does for paper; more than most consuming countries we would benefit from the development of alternative processes. But in the new 'Reports on Applied Chemistry' it is apparent that progress in the year of 1951 did not bring news of any new raw material for paper-making; on the other

hand, it is reported that the use of some of the already known alternatives is 'at last being tackled seriously.' Bagasse is being used for paper-making in Algiers, in the Philippines, in India, and in Brazil. Impressive technical progress is being made in developing straw as a pulp-making material and a consensus of expert and international opinion now holds the view that satisfactory paper can be made from straw. Hardwood pulps, too, are being developed. It must always be to Britain's advantage to encourage these now or improving processes whether or not the different pulp-making materials are distant or accessible. The broader the base of paper supply, the better the world's supply/demand ratio, and as a result the importing of either pulp or paper will not have so exceptionally disturbing an effect upon our general 'terms of trade' equilibrium.

Graded Petrol

THE persistent refusal of the Government to allow the sale of branded petrol in the United Kingdom is much to be regretted, not only from the viewpoint of private and commercial users of road vehicles, but also from the technical aspect which has not, perhaps, been given the publicity or received the consideration it deserves. There are at present six catalytic crackers in the United Kingdom, two in operation and four nearing completion. If used for the purpose for which they were designed—the production of high octane petrol—these should also be yielding large quantities of those gases, such as ethylene and butylene, on which is based the rapidly developing petroleum chemical industry, which is itself a potential dollar saver. From the point of view of the motor industry it is indisputable that premier grade petrol would be beneficial both to the private and commercial users as many light goods vehicles today have the same engines as private cars. Higher octane petrol would enable lower speeds in top gear and an increase in mileage per gallon. The cracking plants (Shell's Stanlow plant cost nearly £9,000,000), are wasted until they can be used for the

purpose for which they were designed, and at present the chemical industry is deprived of a source of possible new chemicals and the motor industry prevented from having the best quality product. Reconsideration by the Government and a public review of all the factors involved are much to be desired.

The Delaney Committee Reports

THE long expected Report of the Delaney Committee on 'chemicals in foods' has now been made. Its moderate tone may come as a surprise to British observers, for the actual hearings in America were accompanied by much more heat and light than is the case with Working Parties or Select Committees here. That, however, is merely one of the differences between American and British ways of life. The final result—the Delaney Committee's third section Report—is a clearly worded and balanced document. It supports the view that the increasing use of chemicals in the production, processing, preservation, and packaging of food has created a serious problem in public health, and it states that existing laws (Federal) do not sufficiently protect the public. The general testimony of witnesses was 'that no chemical should be permitted entry into the nation's food supply until its safety for use has been demonstrated beyond a reasonable doubt.' In practice, this calls for an independent authority or organisation—disconnected with the food and chemical industries—to pass final judgment upon the safety of any chemical additive. No specific methods of test are recommended. It is felt that the nature of tests must depend upon the composition of the chemical and the food involved, together with many other factors such as the breakdown products of the chemical in the food and the amount of such substances already found in normal diets. The onus for showing that a chemical is 'safe beyond reasonable doubt' must fall upon the producers of the food or food products using it. They must submit evidence of safety to the independent authority, who will then decide whether permission for use may be granted.

Special Exclusion Unwise

NEVERTHELESS, there was a minority Report, signed by one member, recommending that pesticides should be accorded a special status excluding them from new legislation recommended for other chemical additives to foods. The basis of distinction would seem to be between chemicals added to foods before and after harvesting. In the majority Report, however, the risk of residues from such applications appearing in final foodstuffs outweighed any handicaps to development and food production that additional legislation might bring. Mr. Horan, the minority dissident, stated that 'the Report is "alarmist" in nature. It would, in my opinion contribute to the difficulties of our producers of foodstuffs in the United States and yet add nothing of assurance to the consuming public.' Mr. Horan presented considerable evidence for his views, particularly along the lines that the toxic hazards of new insecticides are now much better understood than in 1950 or earlier and that existing legislation could safely protect the public. Probably many chemists will incline towards Mr. Horan's arguments. But there is in this matter the 'political' consideration. A great deal of criticism about the toxic risks of pesticides has been voiced and printed. To give these food chemicals a special exclusion from future legislation may in fact produce more public alarm than Mr. Horan fears as the outcome of the Report. If pesticides do not bring measurable risks to public health, the independent authority will surely give them 'clearance.' It should be added that the Delaney Committee had already given fertilisers 'a clean bill of health.'

U.S. Chemical Production Record

Output of industrial chemicals in the U.S.A. reached a new high level in April according to figures compiled by the Federal Reserve Board. Using the average from 1935-1939 as 100, the index production for that month was 567, which showed a gain of 35 points over 1951. No other industrial group approximates such a sharp growth since the war.

Chemistry & the British Association

Programme for Next Month's Belfast Meeting

DOMINANT trends of chemistry in the present and possible developments in the future will be discussed at the 114th annual meeting of the British Association for the Advancement of Science which will be held in Belfast from 3-10 September.

H.R.H. the Duke of Edinburgh, K.G., F.R.S., last year's president, will honour the association with his presence. The first general assembly of members will be in the Sir William Whitla Hall of the Queen's University in the evening of Wednesday, 3 September, when the Chancellor of the University will confer the honorary degree of LL.D. on Professor A. V. Hill, C.N., O.B.E., F.R.S. (this year's president), and Dr. D. W. Bronk, F.R.S. (president of the American Association for the Advancement of Science). Honorary degrees of D.Sc. will also be conferred on Dr. E. D. Adrian, O.M., P.R.S., Sir William Slater and Sir Richard Southwell, F.R.S.

Following the graduation, Professor A. V. Hill will deliver his presidential address on 'The Ethical Dilemma of Science'.

As it is not expected that all who wish to be present will be able to be accommodated in the Sir William Whitla Hall, proceedings will be relayed to the Whitla Hall in the adjacent Methodist College, which will be visited by the Duke of Edinburgh and the president before the graduation ceremony and at the close of the inaugural meeting.

Section B—Chemistry

In Section B—the chemistry section—a diversity of widely interesting papers will be presented. Proceedings of this section will be opened on Thursday, 4 September, by its president, Professor W. Wardlaw, C.B.E., with an address on 'The Advancing Front of Chemistry'. He will be followed by Dr. D. H. R. Barton, who will discuss 'Progress in Organic Chemistry', and Professor A. R. Ubbelohde, F.R.S., who will survey 'Advances in Inorganic and Physical Chemistry.'

Meeting as it is this year in the centre of the world's greatest linen industry area, the chemistry section's session on Friday morning, 5 September, on 'Fibres Old and New' will no doubt prove a special attraction.

The session will be introduced by Dr.

D. W. Hill, of the British Cotton Industry Research Association. Other contributors to this discussion will be Dr. A. J. Turner, C.B.E., director of the Linen Industry Research Association at Lambeg on 'Naturally-occurring Fibres'; Dr. A. R. Urquhart, research superintendent, Lansil, Ltd., on 'Natural-polymer Fibres'; and Dr. Rowland Hill, of Imperial Chemical Industries, Ltd., on 'Synthetic-polymer Fibres'.

Food Processing

'Food Processing' will be the subject of discussion by Section B on Monday, 8 September. After an introduction by Professor H. D. Kay, chairman of the Food Group of the Society of Chemical Industry, the first speaker will be Dr. A. J. Amos on 'The Chemistry of Breadmaking'. Dr. J. A. Lovern, of the Torry Research Station, will follow with a paper on 'Chemistry and Advances in Fish Processing', and the last speaker before the general discussion will be Mr. P. N. Williams, of Unilever, Ltd., on 'The Processing of Fats'.

The last session of the section on the morning of Tuesday, 9 September, will be devoted to 'Biologically Significant Macro-molecules.' This will be opened by Professor M. Stacey, F.R.S., of Birmingham University, with a talk on 'Polysaccharides of Medical Importance'. Dr. B. E. Conway will discuss the 'Physical Chemistry of Deoxyribonucleic Acid', and Dr. G. A. Gilbert's subject will be 'Enzymes for Building Natural and Synthetic Starch'.

Before these proceedings, a special tea party for young men and women chemists under 25 years of age is being arranged by Section B, so that they may have the opportunity of becoming acquainted with the section committee members and other chemists. The section officers are:—*President*: Professor W. Wardlaw, C.B.E.; *vice-presidents*: Professor Wesley Cocker, Dr. A. J. Turner, C.B.E., Professor A. R. Ubbelohde, F.R.S., Professor T. S. Wheeler, and R. Clement Wilson; *recorder*: Professor L. Hunter, University College, Leicester; *secretaries*: Dr. J. Dewar and Dr. D. C. Martin; *local secretary*: Dr. R. C. Pink.

Chemical engineering has its share of dis-

cussion in the Engineering Section (G), when Chemical Engineering in Industry will be the subject of Session B on the morning of Monday, 8 September. Professor D. M. Newitt, M.C., F.R.S., Courtauld Professor of Chemical Engineering, Imperial College, London, will speak on 'The Training of Chemical Engineers', and Dr. E. H. T. Hoblyn, director, British Chemical Plant Manufacturers' Association, will deliver a paper on 'The Chemical Engineer in Industry'. The general discussion will be opened by Sir Harold Hartley, K.C.V.O., C.B.E., M.C., F.R.S., past-president, Institution of Chemical Engineers, president of the British Association, 1950.

The Exchange Lecture of the British and American Associations for the Advancement of Science will be delivered by Dr. D. W. Bronk (president of the American association), in the Sir William Whitla Hall, Queen's University, at 5.30 p.m. on Thursday, 4 September.

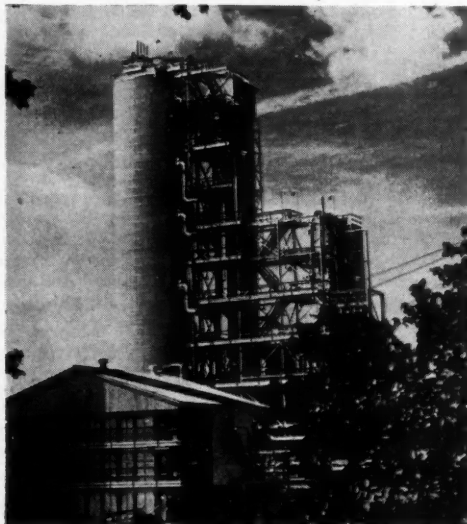
As usual, in connection with the meeting, a variety of visits have been arranged to local industrial and other places of interest. These are fully set out in a special Excursions Guide prepared in Belfast and consist of general excursions of interest to all members and sectional excursions which are mainly of interest to specialists.

The general programme includes a number of social functions, a series of five lectures for young people and three evening discourses. The evening discourses, which will be delivered in the Sir William Whitla Hall, at Queen's University at 8 p.m., will be as follows:—

Friday, 5 September: 'Oil in Peace and War' by Mr. A. C. Hartley, C.B.E., past-president, Institution of Mechanical Engineers. Sunday, 7 September: 'A Plea for Heretics' by Professor A. Macbeath, Department of Philosophy, Queen's University, and Monday, 8 September, 'Science and an Expanding Agriculture', by Sir William Slater, secretary, Agricultural Research Council.

Too Few Chemical Engineers

Although the number of chemical engineers in the U.S.A. has been tripled in the last decade (1940-1951) the present total of 45,000 is not sufficient to meet the increasing demands being made on the youngest of the major branches of engineering. This is the conclusion of a survey of about one-third of all chemical engineers, and one-third graduate students of chemical engineering carried out by the U.S.A. National Scientific Register, with the aid of the American Chemical Society.



Great progress is being made in the construction of the Anglo-Iranian Oil Company's Kent Oil Refinery, Isle of Grain. The photograph on the left is of the vast distillation unit which will process 4,000,000 tons of crude oil a year

Sulphur & Pyrites in Canada

Development of Resources Governed by Economic Recovery

FOR many years, Canada has depended upon cheap readily-available Frasch-mined sulphur from the U.S.A. However, with the marked industrial expansion of recent years and the rapid depletion of reserves this source can no longer be expected to meet all the requirements of the Dominion.

Canada, like most industrial countries, is being forced to seek other sources of supply. Fortunately, few technical difficulties exist since practical processes for sulphur recovery are available for the main alternative sources. The element, in various forms, can be recovered from base metal smelting operations, natural gas, bituminous sands, stack gases, anhydrite and pyrites deposits. Of these, there is an abundant supply in Canada.

The problem which confronts industry is primarily one of economics and convenience. Under present conditions, the alternative sources cannot compete with Frasch-mined sulphur. However, a continuing scarcity of brimstone will make the exploitation of these sources commercially feasible. Already, several important projects for the recovery of by-product sulphur are underway and others are in the proposal stage.

A report 'Sulphur and Pyrites in Canada', by T. H. Jones, engineer, Industrial Mines Division, has now been issued as Memorandum Series No. 118 (April, 1952), by the Mines Branch, Ottawa, of the Department of Mines and Technical Surveys, Canada.

This report reviews production and consumption in Canada and describes possible alternative sources of domestic supply as well as the vast reserves of pyrites which are known to exist.

Concise Summary

In order to give a clear picture of the sulphur problem, a concise summary is given of world production and consumption, shortage, and the difficulty of finding new salt-dome deposits, before dealing specifically with the Canadian aspects.

Discovery of a new sulphur deposit by the Texas Company while drilling for oil on the Gulf Coast in Louisiana was announced by Freeport Sulphur Company in 1951. It is

expected that this deposit will require an investment of from \$10,000,000 to \$15,000,000 to produce at the rate of 500,000 tons a year by the end of 1953.

Several other domes are being prepared for production in Louisiana and Texas. However, production and consumption of sulphuric acid has been rising at a rapid rate over the past several years and if civilian and defence requirements continue to rise at the same rate, the sulphur supply situation is likely to remain 'tight' for a long time to come.

Tightening Up Expected

It has been estimated that the U.S.A. will need 8,000,000 tons yearly of elemental sulphur by 1960. If this proves to be correct it seems evident that the supply of brimstone will be more rigidly allocated in 1960 than it is now unless the use of other sources of sulphur is greatly expanded in the meantime. (*Canadian Chemical Processing*, June, 1951).

There are no elemental sulphur deposits known to exist in Canada comparable to those of the Gulf Coast region of the U.S.A. All production has been based on alternative sources. In the past, production was limited to pyrites from Canadian deposits; at present, to by-product pyrite and smelter gases.

From 1915 to 1918, the total Canadian production of sulphur was derived from pyrites, or pyrrhotite, obtained from the Eustis and Weedon mines in Quebec; the Guodreau Lake, Northpines, Sulphide, Queensboro, Craig and Bannockburn mines in Ontario and the Anyox and Sullivan mines in British Columbia. Ontario was the major producer with Quebec second and British Columbia third.

No mines operate in Canada, today, solely for the production of pyrites, since it cannot be mined profitably in competition with elemental sulphur from the U.S.A.

All the pyrite which is produced in Canada, at present, is obtained as a flotation by-product of base metal mining operations. Such production has been considered a small-profit operation bringing only from \$2.00 to \$2.50 per short ton, f.o.b. the mine, to the producers.

In 1950, it was produced by the Noranda, Waite, Amulet, Quemont and East Sullivan mines in western Quebec and by the Britannia mine in British Columbia. The production amounted to a sulphur equivalent of 150,828 tons. Britannia mine is operated by Britannia Mining and Smelting Co., Ltd., a subsidiary of Howe Sound Company of New York. The mine has been a regular contributor to Canada's output for many years. The pyrite is obtained as a by-product from the copper-gold-silver operations at Britannia Beach, B.C.

Two-Thirds Exported

About 65 per cent of the by-product is exported. Nearly all of this goes to the acid manufacturers in the U.S.A. Most of the remainder is consumed in the acid plants of Nichols Chemical Co., Ltd., at Valleyfield, P.Q., Sulphide, Ontario and Barnet, B.C. The rest is used by St. Lawrence Paper Mills, Ltd., in its sulphite mill at Three Rivers, P.Q. The latter is the only paper company now burning pyrite as a source of sulphur.

Consolidated Mining & Smelting Company of Canada, Ltd., at Trail, B.C., converts the sulphur dioxide to the smelter gases to sulphuric acid. The entire output is used to manufacture fertiliser at its nearby plant.

The only other Canadian production from smelter gases is at the sulphuric acid plant adjoining the Copper Cliff smelter of the International Nickel Company of Canada. This plant, owned by Canadian Industries, Ltd., has been in operation for several years. It has produced from 35,000 to 45,000 tons of 100 per cent sulphuric acid (12,000 to 15,000 tons sulphur equivalent) annually.

Major expansion is underway. By 1953, the plant is expected to produce 90,000 tons of liquid sulphur dioxide annually for the pulp and paper industry.

Deficiency in domestic production will probably exist for many years but projects are at present underway in Canada, which make the long term outlook encouraging.

Sulphuric acid production in Canada began in 1867 when a plant was built in London, Ontario. It manufactured acid from brimstone to supply the needs of the infant oil refining industry in south-western Ontario. The second plant to manufacture sulphuric acid was built in Brockville, Ontario, in 1869 and burned pyrite from eastern Ontario mines as the source of sulphur.

Since 1867 about 20 acid plants have been

built and operated in the Dominion with 11 of them burning brimstone and the remainder burning pyrite or making use of the SO_2 content of smelter stack gas as the source material.

There are at present nine plants in Canada manufacturing sulphuric acid of which four use brimstone as the sulphur source, three use pyrite obtained as a flotation concentrate from the milling of base metal ores, and two use sulphur dioxide contained in the stack gases from the smelting of base metal ores or concentrates.

In addition to the plants now manufacturing sulphuric acid several other plants are either in the construction stage or are being contemplated. All of these projects will use material from Canadian sources.

Studies have been made, by several firms, of the possibility of recovering the sulphur dioxide or hydrogen sulphide content of the gas streams of oil refineries in Canada. The largest concentration of refining facilities in Canada is located in Montreal East where Imperial Oil, British American Oil, Shell Oil, and Texaco Oil companies have a combined throughput of about 150,000 barrels a day.

Most of the crude refined in these plants comes from Venezuela via pipeline from Portland, Maine, and has a very low sulphur content (under one per cent). Minor amounts of Middle East crudes are consumed. They contain up to five per cent sulphur.

One-Half Recoverable

Total content of sulphur in the gas streams from the four refineries has been estimated at about 40 tons per day. Half of this is considered recoverable. Several factors, however, tend to prevent the treatment of gases from this group of refineries:

1. The gathering of gases from several sources, some distance apart, would be difficult.
2. The extreme climatic conditions in the area would make capital outlay, with respect to collection and transmission of gases, high.
3. The relatively low H_2S content of the gases would preclude the economical recovery of the sulphur content as brimstone.
4. In some cases, the refineries burn their waste gases for preheating the crude and would require payment for the H_2S , contained in the gas, on a B.T.U. basis.
5. The end product—sulphuric acid—

must be marketed in a highly competitive area and the economics of producing and marketing a relatively small output of sulphuric acid (50-60 tons per day) would not be worthwhile.

Elemental sulphur, or brimstone, was produced, from 1936 until 1943, by the Consolidated Mining and Smelting Company of Trail, B.C. This operation was discontinued in 1943 when production facilities were switched back to production of sulphuric acid from the stack gases.

A proposed plant of Noranda Mines, Ltd., in which 300 tons of pyrite would be roasted daily, would result in the annual recovery of about 18,000 tons of brimstone, and 35,000 tons of sulphur equivalent as sulphuric acid. Plans for the construction of the plant are being held in abeyance and no official announcement has been made by company officials on the beginning of plant construction. A ready market exists for any brimstone produced but it would appear that the marketing of large tonnages of sulphuric acid presents a problem.

Recovery of sulphur, in elemental form, from 'sour' natural gases containing hydrogen sulphide is a development of recent years. The amount of H_2S in natural gases varies with the field but large volumes of proven reserves have been established in the Pincher Creek, Jumping Pound and Turner Valley fields of Alberta. These are estimated to contain, eight, four, and two per cent hydrogen sulphide gas respectively. Approximately 44.6 tons of elemental sulphur are contained in 1,000,000 cu. ft. of H_2S gas. A recovery of 80 to 90 per cent is possible.

Combined Effort

Shell Oil Company of Canada, in conjunction with Powell River Company of British Columbia, a large producer of newsprint, is building a \$500,000 sulphur recovery plant in the Jumping Pound field of Alberta to recover brimstone from the scrubbing of sour natural gases. The gas scrubbing plant, officially opened in May, 1951, cleans about 25,000,000 cu. ft. of natural gas daily and upon completion of the sulphur recovery plant in 1952, about 30 tons of brimstone will be recovered each day. The H_2S in the natural gas is first extracted with amines and one-third of it is oxidised to form SO_2 . The SO_2 and H_2S gases react in the presence of an alumina catalyst to yield elemental sulphur.

The Canadian Western Natural Gas Company distributes the scrubbed gas output to Calgary and Southern Alberta consumers.

Construction of a \$350,000 sulphur recovery plant adjacent to its present gas scrubbing plant in the Turner Valley field of Alberta has been announced by the Royalite Oil Company. The plant is scheduled to be in operation by mid-1952 and will have a daily capacity of 30 tons of brimstone.

Overall recovery of sulphur in this type of plant is reported to be about 90 per cent and the sulphur produced is comparable in quality to that of Frasch-mined sulphur.

Sour Natural Gases

Naturally, the amount of elemental sulphur recoverable from sour natural gases depends entirely on the volume of gas consumed and its H_2S content. Within the next few years, possibly some 50,000 tons of brimstone might be recovered annually but in the immediate future this will be considerably less.

A huge potential source of sulphur in Canada exists in the extensive deposits of bituminous sands along the Athabaska river in northern Alberta. The deposits are about 200 to 230 air miles north of Edmonton. Several different processes for the extraction of the bitumen from the sand have been developed by the Alberta Government, the Fuels Division of the Department of Mines and Technical Surveys at Ottawa, and others.

Natural sulphates—anhydrite and gypsum—are not at present important sources of sulphur in North America because of the economic difficulties.

The large deposits of sodium sulphate, which occur in southern Saskatchewan and Alberta, constitute a large potential source of sulphur. By using processes already known the sodium sulphate can be considered as an important possible source of soda ash and elemental sulphur.

One process consists of heating, by coal, gas, or oil the sulphate mixed with lignite coal in a rotary kiln to about 800°C. The sulphate is reduced to a liquid melt containing sodium carbonate and sulphide which is run directly into water. Carbon dioxide is bubbled through the solution and changes all the sodium into carbonate form from which it can be recovered as soda ash. The sulphuretted hydrogen (hydrogen sulphide) evolved can be converted by the Chance-

Claus process to a high purity elemental sulphur.

This conversion step is identical to that which will be used by Shell Oil Company and Royalite Oil Company to recover elemental sulphur from sour natural gases in Alberta. If such a process were established in the Canadian West to recover soda ash and sulphur it would provide outlets for sodium sulphate, oil or gas, and lignite coal and could go far to overcoming the shortages of both soda ash and sulphur in the Dominion.

'Ferralyst' Process Described

In dealing with the recovery and utilisation of sulphuric acid from H_2SO_4 , the report describes separation from oil refinery sludge and recovery from spent liquor. It then goes on to describe the 'Ferralyst' process for the production of elemental sulphur from waste or natural gases and quotes extensively from THE CHEMICAL AGE (65, 143), in which the method was first exclusively described in the U.K.

The problem of increasing domestic supplies in Canada is limited to the utilisation of sulphur compounds for conversion into brimstone, liquid sulphur dioxide and sulphuric acid. Since practical processes exist for these conversions and since the Dominion has large resources of sulphur compounds, the problem resolves itself around economics and the availability of markets for the end-products.

Largest consumer of sulphur in Canada is the pulp and paper industry. It needs sulphur in a form which can be used in producing sulphur dioxide for its sulphite plants. With the exception of one mill which burns by-product pyrite, the needs of the industry are imported as brimstone from the U.S.A. Capital costs of pyrite-burning plants are estimated to be three times those of sulphur-burning plants. Furthermore, elemental sulphur is preferred because of the convenience of handling, storage, disposal of waste and quality of the finished product.

Due to the uncertainty of future supplies of brimstone, several pulp and paper companies in Canada are considering burning pyrite rather than brimstone as a source of sulphur dioxide gas.

Current contract price paid to producers of pyrite concentrate does not justify opening up long-dormant properties for the production of pyrite grading 48 per cent, or

better, sulphur. A price of at least 10 to 12 cents per short ton unit of sulphur, f.o.b. mine, would be necessary to induce mining companies to 'go after' pyrite.

Recovery of sulphur dioxide, as a liquid by Canadian Industries, Ltd., from stack gases at Copper Cliff will alleviate the sulphur problem of some pulp and paper mills. The production—about 45,000 tons equivalent sulphur—would supply about 15 per cent of the total requirements of the industry. It will not be available until late 1952. Freight haulage distance will always be a limiting factor in the use of liquid sulphur dioxide by the pulp and paper industry.

A process developed by Noranda Mines, Ltd., for the recovery of elemental sulphur from a typical, finely ground pyrite flotation concentrate, is not yet committed to commercial plant operation. It is estimated that 18,000 tons of elemental sulphur might be produced annually from a 300-ton per day plant if commercial operations are undertaken. Only a third of the sulphur content of the pyrite is recoverable as elemental sulphur with the remainder recoverable as sulphuric acid.

The other product of the process is high-grade iron oxide sinter. Ready markets for sulphuric acid and iron oxide sinter would be necessary for economic operation of the process.

Quantities Regulated

Quantities of sulphur recoverable from sour natural gas are regulated by its hydrogen sulphide content and the volume of gas cleaned. As stated before, it is not likely that production from this source would reach even 50,000 tons of elemental sulphur annually, for several years.

Recovery of sulphur from oil refinery, coke-oven and other industrial gases (it is said) is not likely, if ever, to enter very widely into the Canadian sulphur picture. Recovery of sulphuric acid and Portland cement from anhydrite or gypsum deposits in Canada appears to lie at some future date.

Finally, the huge potential of sulphur which exists in the bituminous sands of Alberta will only be exploitable should oil extraction plants and refineries start operating.

No single, quick solution presents itself but rather a variety of programmes will each contribute to the solution of the Canadian sulphur supply situation in years to come.

A Review of Phenol Analysis

Part IIIc—Determination in Tars & Allied Products

IN the last part of this review (see THE CHEMICAL AGE, 67, 181), the quantitative determination of phenol in biological materials was reviewed. This part will be devoted to its quantitative determination in tars and allied products.

(a) TARS: Traces of phenol in bitumen, asphalts and tars may be determined satisfactorily using the Folin-Denis and the Hinden classical methods. Kunzler and Hess⁷⁷ reported this after a critical study of available methods for the determination of phenols, with particular reference to cable-impregnating materials. The phenols were first separated as follows:—Mix 20 g. of impregnating material or cable covering, containing paper and jute, but no stones and earth, with 10 g. of copper sulphate, 2 ml. of 1:4 phosphoric acid and 20 ml. of distilled water. Pass steam through the sample contained in a copper coil, and after the extraction condense the distillate in a copper pipe until 2-2.5 litres are collected.

The method of Chapin was found to be quite insensitive for the determination, and the method of Houghton and Pelly was not satisfactory. Only 2,4-dinitrophenol, of 15 phenolic compounds tested, gave no reaction with any of the methods examined. The others could be determined in concentrations of 0.1-1 mg. per litre. Samples from a 900 mm. length of corroded cable showed varying phenolic contents depending on the colour reaction used, as well as on the soil conditions to which parts of the cable had been exposed. Most of the cable-impregnating materials investigated by the authors appeared to contain phenols, the amount varying from 0.0001-1.5 per cent.

Water Soluble Phenols

Feil⁷⁸ has adapted the Millon colorimetric procedure for the determination of water-soluble phenols in tars or tar mixtures. The method consists essentially of adding to 50 ml. of liquid tested, exactly 0.5 ml. of 2N nitric acid and then 0.2 ml. of Millon's reagent, heating to the boiling point, keeping the liquid 1 minute at this temperature without boiling, and cooling and comparing its colour with water by a Pulfrich photometer.

Current methods for the determination of

phenols in tars and creosotes have been examined by Verhulst⁷⁹. Where speed is important, phenols can be analysed by the old method of absorption with sodium hydroxide solution and measuring the change in volume. Verhulst describes a gravimetric method to be used directly on creosotes and primary tars without involving a distillation process. The phenols are extracted by washing with concentrated sodium hydroxide solution, and are then washed with ether. The phenols are released with hydrochloric acid, extracted with ether, the ether evaporated in a weighed flask, and the phenols weighed. The method distinguishes between lower phenols and resinous phenols.

Gravimetric Determination

Chopin⁸⁰ has determined gravimetrically the phenol in tar or distillate:—Weigh out accurately 8-9 g. of sample, and transfer from the weighing dish to a separating funnel by means of 50 ml. of carbon tetrachloride. Add 100 ml. of 10 per cent aqueous sodium hydroxide and agitate the mixture for 30-40 minutes. Decant off the solution during 15 minutes, wash the aqueous solution of phenates with 50 ml. more of carbon tetrachloride, and decompose by adding 30-40 ml. of 6N hydrochloric acid. To extract the phenols from this mixture, add 20 ml. of carbon tetrachloride or chloroform. Agitate for three minutes and withdraw the organic solvent layer through a glass filtering crucible. Repeat this operation five times and distil until the phenols show a tendency to come over. Eliminate moisture by adding 10 ml. of benzene and distilling again. Cool and weigh the residual phenols. A titrimetric procedure can also be applied: Place 7-8 ml. of dry tar (of known density) in a separating funnel, and warm to 40°C. until fluid. Add 10 per cent aqueous sodium hydroxide in volume equal to that of the sample. Add 30 ml. of dry carbon tetrachloride, shake for at least 30 minutes and allow to stand until two distinct layers are formed. Draw off the bottom layer, and repeat the treatment with fresh solvent. Read the volume of carbon tetrachloride before and after the treatment.

A new titrimetric method for the exact

determination of phenols in low temperature tar and tar oils has recently been outlined by Lambris and Haferkorn¹¹. A 3 g. sample of water-free tar or tar oil containing approximately 5 per cent of phenols is dissolved in a mixture of benzene and xylene, and a known excess of a 20 per cent solution of potassium hydroxide (of known normality and saturated with benzene and xylene) is added. The weight of the potash is determined by difference.

Hot Water Extraction

This mixture is shaken repeatedly in a 300 ml. separating funnel. After standing for 30 minutes, the dark-coloured phenolate solution containing the major portion is separated and weighed. Care must be taken to prevent the introduction of solids. The phenolate in the residue is extracted with hot water and titrated with 0.2N hydrochloric acid using 1 ml. of 1 per cent Congo red as indicator. If water is present in the tar or tar oil, 100 ml. of xylene is added immediately after weighing, and the water separated by distillation, its weight being determined. Any phenols carried over are dissolved in the small amount of xylene in the distillate. This amount is added to the bulk of the xylene. After any remaining phenols are extracted from the tar residue with boiling benzene, the benzene-xylene mixture is treated with potash as above. The accuracy of the method is estimated to be ± 1 per cent as shown by experiments with phenol, *o*-, *m*- and *p*-cresol, and catechol. The weight of the dissolved phenols, x is determined by the equation:

$x = c - a + cd/ab - d$ where ' a ' = weight of potassium hydroxide; ' b ' = hydrochloric acid used per g. of potassium hydroxide; ' c ' = weight of major portion of phenolate solution which is formed by shaking the phenol solution with potassium hydroxide; ' d ' = hydrochloric acid used for titrating the phenolate residue.

Physico-chemical methods of tar analysis are extensively used. Masse and Leroux¹² used a freezing-point technique to determine phenol in the crude phenols found in coal tar. They found that a mixture containing 67 per cent of phenol and 33 per cent of *o*-cresol solidifies at 21° C. Similarly, mixtures containing 67 per cent of phenol, and 33 per cent of a mixture of 60 parts of *m*-cresol and 40 parts of *o*-cresol, and of 67

per cent of phenol and 33 per cent of a mixture of 50 parts of a mixture of 50 parts of *o*-cresol and 50 parts of the 60 : 40 mixture both solidify at 21°. The freezing-points containing intermediate concentrations of phenol lie on a straight line (plotted by using the percentage of phenol and the freezing-points as axes). Provided the cresols present do not exceed 33 per cent of the mixture, the percentage of phenol in a cresol-mixture may be fairly accurately calculated from the freezing point of the mixture.

The following is an outline of the method as applied to coal tar:—3 Kg. of the crude phenol fraction are fractionally distilled from a copper flask fitted with a long Vigreux column, 3 fractions being collected (i) water and phenols (B.P. < 180° C.), (ii) phenols (B.P. 180°-203° C.), (iii) phenols (B.P. > 203° C.). Addition of solid sodium chloride to (i) causes the separation of dissolved phenols which are separated from the water and combined with (ii). Fraction (iii) is used simply to rinse out vessels, after (i) and (ii) have been returned to the distilling flask. The combined phenols are then redistilled and fractions of 250-300 g. each collected until the temperature reaches 198° C. The freezing-point of each fraction is then separately determined and the percentage in each calculated. (If in any case the freezing-point falls below 21° C., known amounts of commercial phenol are added to bring the percentage of phenol up to 67 per cent.

Volumetric Method

A volumetric method, using glycerol as solvent, has been devised by Lugovkin¹³ for the determination of phenols in distillation tars. The method is based on the observation of Fisher and Gluud that glycerol dissolves phenols to the exclusion of hydrocarbons. The method is rapid, and gives exact results, but it can only be used when paraffins are not present in the distillates. The presence of the usual amounts of moisture (8-15 per cent) does not interfere; only when the water content reaches 60-70 per cent is the extraction of phenols incomplete. The distillation tar is introduced into a 35 ml. graduated cylinder, an approximately equal volume of glycerol (S.G. 1.245 g. at 25°) added, the cylinder stoppered, and after standing 2-3 minutes (or on being immersed in a hot water-bath) the

contents are separated into two layers. The upper layer consists of neutral substances and the lower of the solution of phenols in glycerol. When operating with viscous distillates, the glycerol should previously be diluted with kerosene. The percentage of phenols is calculated from the decrease in the volume of the tar layer. The extracted phenols may be recovered by precipitation from the glycerol solution by diluting the latter with water.

Ubal dini and Mochi¹⁴ have claimed that existing procedures for the determination of phenols in tars may give results varying from 20-30 per cent. An improved method is to put 20 g. of tar in a vacuum-walled vessel, add 10 ml. of sodium hydroxide solution ($d = 1.332$), the tar and sodium hydroxide solution being at the same temperature before mixing. The two are mixed, stirred with a glass rod, and the maximum temperature reached during the reaction is observed with a thermometer reading to 0.1°C . The maximum temperature is reached a very few seconds after mixing, and lasts long enough for reading. Dividing the rise in temperature by 0.42 gives directly the percentage of phenols present.

(b) **TAR OILS:** Both crystalline phenol and *m*-cresol have been determined in crude phenol oil.¹⁶ 1 Kg. of oil is stirred for 2 hours with 1½ litres of an alkaline solution containing 150 g. of sodium hydroxide, and left to settle for 2 hours, after which the phenols are separated from the oil. If naphthalene crystallises in the intermediate layer, it is filtered by suction, and the phenates in the filtrate, are separated from the oil and added to the main portion of phenates. The phenates are steamed with 1 Kg. of direct steam, and on cooling are decomposed with 400 ml. of concentrated hydrochloric acid (S.G. 1.19). After allowing the phenols to settle for 30 minutes, the mother liquors are poured off, treated with 300 g. of sodium chloride and left overnight to precipitate the rest of the phenols. The crude phenols thus obtained are subjected twice to fractionation.

Two Fractions Obtained

Two fractions are finally obtained, one boiling up to 195°C . and the other between 195°C . and 207°C . The first fraction contains almost all of the crystalline phenol which is determined by the Masse-Leroux method with slight modifica-

tion. In the second fraction, *m*-cresol is determined by Raschig's method, which consists in nitrating to obtain trinitro-*m*-cresol. If the fraction contains a high percentage of *m*-cresol, Raschig's prescriptions must be modified, and the nitric acid used for nitration must in that case be about 80° strong in order not only to obtain more exact results, but also to have trinitro-*m*-cresol in fine crystalline structure which facilitates uniform drying.

Nitroso-Phenol Reaction

The nitroso-phenol reaction has been applied to the colorimetric determination of phenols in petroleum oils.¹⁸ The method employs sodium nitrite in place of the nitric acid formerly used. The nitrosophenol formed rearranges in the presence of ethanol and ammonia to form a highly coloured quinonoid radical. The nitroso-phenol, in the new method, is formed at room temperature under optimum conditions, and the colour is measured in a photo-electric colorimeter. The sample is treated directly with a buffer solution containing 800 ml. of glacial acetic acid, 150 ml. of 10 per cent potassium hydroxide solution and 50 ml. of water, or, if the material is insoluble in the buffer solution, it is treated with the potassium hydroxide and a suitable diluent. The phenol goes into the lower layer in the separating funnel. The extract is then treated with acetic acid in excess, followed by a little concentrated sulphuric acid and a little saturated sodium nitrite solution. After a short reaction period, a solution prepared from 450 ml. of ethanol or propanol, 300 ml. of ammonia (0.88) and 250 ml. of water is added. Enough of this solution is added to make the total volume 50 ml. After standing at least an hour (preferably overnight) a colorimeter reading is obtained at 420 μ . The method is not affected by organic compounds containing other functional groups, or by water, mineral acids, inorganic bases or inorganic salts. The results are usually within 1 per cent of the truth, if standards are chosen containing phenol material which corresponds closely to that of the sample analysed.

Freezing-point and specific gravity measurements have been employed by Weiss and Downs¹⁷ in an accurate method for the determination of phenol in crude carbofic acid and tar oils. The principles of the method are as follows:—The dirty oil is

distilled, taking the total distillate from start to coking, and a weight of oil, which contains between 300 and 350 g. of acids, is treated in a separating funnel with sodium hydroxide solution. After settling is complete, the phenolate is separated, and the oil washed several times with alkali. The separated alkali solutions are added to the first phenolate, which is then extracted several times in a separating funnel with 100 ml. portions of pure benzene. After separation of the benzene, the phenolate is run into a beaker and brought to a gentle boil to expel the benzene. After cooling, the purified phenolate is placed in a separating funnel and acidified with a slight excess of 25 per cent sulphuric acid, keeping the temperature of the solution below 40°C. Complete separations occurs after 3-4 hours, and the sulphate layer is then drawn off and kept for further treatment, while the acid layer is drawn off into a weighed 500 ml. round-bottomed flask. The sulphate solution is extracted three times with pure benzene, and the benzene extracted with sodium hydroxide solution.

Acid Layer Separated

After drawing off all the benzene from this and acidifying with sulphuric acid, the acid layer is separated and added to the flask containing the main portion of the acids. After weighing the flask, the acids are fractionally distilled at the rate of 1 drop per minute. The fractions 179°-190°C. and 190°-202°C. are collected, and the latter is redistilled, after drying the condenser tube, using the weighed flask containing the <190° fraction from the first distillate as a receiver. The distillation is continued up to 197°C., and the receiver containing the combined fractions up to 197°C. is then weighed and stoppered. The fraction is then tested for specific gravity and freezing-point, and the values found are compared with standard phenol determination curves.

Petrie²⁸ has compiled some valuable notes on the determination of phenol in tar oils. His experiments on methods in use at that time showed that the methods (loc. cit.) of Fox and Barker and Weiss and Downs compared favourably, the latter giving slightly higher results. The paper deals with modifications of the quicker method, that by Fox and Barker. Calcium chloride, plaster of Paris and anhydrous sodium sulphate are

not satisfactory agents for drying phenolic mixtures. Petrie eliminates the water with benzene by distillation and careful exclusion of moisture afterwards. The modified method is:—

Dissolve the mixture in twice its volume of benzene and extract with 10 per cent sodium hydroxide solution, using first 400 ml. and then 100 ml. Allow the phenolate solution to settle for 30 minutes, run off, and distil with steam. Cool, transfer to a 2-litre separating funnel and liberate the phenol acids by means of dilute sulphuric acid (1:1), keeping the liquid cool. Allow to stand for 4 hours, separate the sulphate liquor, and transfer the phenol acids to a 300 ml. separating funnel. Extract the sulphate liquor with three 30 ml. portions of benzene, and add the extracts to the phenol acids. Discard the water which separates. Transfer the liquid to a 300 ml. round-bottomed flask, and fractionate over a 12-inch head. Water and benzene distil over at about 75°C., then almost water-free benzene. The first 2-3 drops of phenol distilling at 180°C. are discarded, if contaminated with benzene or water. Change the condenser and collect the distillate to 195°C. in a tarred apparatus from which moisture is excluded. Mix the distillate with pure phenol and read off the percentage of phenol from the curve.

Iodometric Procedure

An iodometric procedure for phenols and cresols has given satisfactory results in the analysis of creolin and similar products²⁹. The phenolic material is prepared for titration as follows:—Weigh out 5 g. of creolin, dilute with 100 ml. of warm water, and add 20 ml. of 10 per cent sodium hydroxide solution; shake, extract three times with ether, evaporate the ether, add water to the residue until a clear solution is obtained, acidify slowly in the cold until a resinous precipitate appears and then add an excess of cold barium chloride solution. Treat the mixture with a saturated solution of barium hydroxide until slightly alkaline, filter, wash with water, and evaporate the filtrate on a water-bath to about 200 ml. Acidify with hydrochloric acid, and distil $\frac{1}{3}$ of the contents, receiving the distillate in 20 ml. of 10 per cent sodium hydroxide solution. Add more water to the flask and distil again, collecting all distillates in a 250 ml. graduated flask. Make up to volume with distilled water,

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shake, withdraw 10 ml. into a beaker and heat to above 60°C.

To carry out the titration, add 25 ml. of 0.1N iodine solution, stand for 10 minutes, acidify with dilute sulphuric acid, dilute with water and let the precipitate stand for a short time. Then decant the liquid into a 500 ml. beaker, filter the precipitate, wash, and add the filtrate to the part decanted. Titrate the excess of iodine with 0.1N sodium thiosulphate solution in the usual way. If N is the amount of thiosulphate used in ml., $25N \times 1.012$ = percentage of phenols + cresol in the creolin.

Pokrovskaya¹⁰ also prefers an iodometric method for the determination of phenols in tar oils. A measured quantity of sample is placed in a 200 ml. measuring flask. Add benzene or toluene to the mark, shake, transfer a portion of the solution to a 250 ml. Erlenmeyer flask and add 5-25 ml. of 0.1N iodine solution, and the same quantity of 0.1N sodium carbonate solution. The quantities of oil and iodine used depend on the phenol content of the oil: the excess of iodine during the reaction must be approximately 100 per cent. Shake the mixture for 10 minutes, add 1-5 ml. of N sulphuric acid and 50-100 ml. of water, and titrate the excess iodine with 0.1N sodium thiosulphate solution. The volume percentage of phenols in the oil is $100 \text{ aK} / 1.04$ ('a' is the amount of 0.1N iodine solution in ml. used per ml. of oil; K is the quantity in g. of phenols reacting with 1 ml. of 0.1N iodine solution, and 1.04 is the density of the phenols). K is determined empirically.

Skirron¹¹ determined phenol in coal tar oils by fractionating about 1 litre of sample, determining the oxygen absorption of the fractions by means of potassium permanganate solution, and calculating the percentage of phenol from the tabulated oxygen absorptions of pure phenol.

Improvements Suggested

Morgan and Meighan¹² have critically reviewed the classical methods for the determination of phenols in tar oils and have advanced some improved methods of analysis. They describe, for example, a new and accurate method for determining both the sodium hydroxide equivalent of tar acids in tar oils, and the formula weight of phenols:—About 2 g. of granular sodium, contained in a 25 mm. side-necked test-tube attached to a water-jacketed gas burette, is

covered with 15-20 ml. of toluene, which is boiled gently to dry the toluene and drive the air out of the tube. After cooling to room temperature, the weighed sample of tar oil or phenol in a small bottle is dropped into the toluene, where the phenol is acted upon by the sodium, precautions being taken against a too-rapid reaction. The hydrogen gas evolved is measured in the gas burette, a correction for toluene vapour being made. The data to be collected and method of calculation, from the hydrogen liberated, necessary to express results in sodium hydroxide equivalents are given. For the success of the method, the phenolic solution must be thoroughly dry.

Modified Method

In the modified method for determining the phenols in tar oils, the tar acids are extracted by shaking with sodium hydroxide solution. The sodium phenolate solution is drawn off into an Erlenmeyer flask, acidified with 20 per cent sulphuric acid, cooled to room temperature, then poured through 65 ml. of benzene in a tar acid funnel (type 2). The sulphate solution is drawn off into the original flask used, to rinse it, then poured back through the benzene. Sufficient benzene is added to bring the total volume to 100 ml., then 50 ml. of 10 per cent sodium hydroxide solution is added and the funnel is thoroughly shaken. The sodium phenolate solution is drawn off and the treatment repeated with 20 ml. of 10 per cent sodium hydroxide solution. The decrease in volume of the benzene is taken as the volume of tar acids, and if a 100 ml. sample of oil is used, this shrinkage in volume represents the percentage by volume of tar acids present in the oil.

A volumetric technique was also preferred by Pontio and Gandin¹³ for the determination of phenols in creosote oils and pitch. The phenolic constituents (phenols and the three cresols) and naphthalene constitute the substances which are of importance in connection with the preservation of wood. These are best determined in the fraction that boils between 125° and 260°C., a much wider temperature range than would appear necessary, perhaps, from the boiling points of the individual constituents. The four phenols can be extracted by three treatments with 100 ml. of 50 per cent acetic acid. From the acid extract, they can be removed by chloroform extraction of the nearly neu-

tral solution, and the phenols can be removed from the chloroform by shaking with 20 ml. of 20 per cent sodium hydroxide solution. After reprecipitating the phenols with hydrochloric acid, and allowing to stand for 24 hours, the volume can be read in a calibrated tube. To purify, dissolving with a mixture of ether and petroleum ether, extracting with sodium hydroxide and reprecipitating with hydrochloric acid, is recommended.

Pieters²⁴ has compared four methods of determining phenol in tar oils: all are based on the volume change of a sodium hydroxide solution after shaking it with the oil. For high phenol content, all methods are equally good: for low phenol content, the official method of the Dutch State Mines is recommended. In a 250 ml. stoppered, graduated flask, heat 50 ml. of 30 per cent sodium hydroxide to 60°C., and read the volume. Add 100 ml. of benzene and the distillate from 100 ml. of the tar oil. Shake thoroughly, again place in 60°C. water-bath and read the volume of the alkali. The increase minus the water content of the oil is the percentage of phenol. A good separation of oil and alkali takes 1 hour. The method gave the highest results of the four used on samples of lignite tar and other tar oils.

Phenols in Petrols

Phenols in petrols and cresylic acids can be determined²⁵ by a u.v. spectrophotometric technique, using the absorption band at 290 m μ . The phenols are extracted with an aqueous solution of sodium hydroxide. An empirical specific extinction coefficient of 24 was used after investigation of some pure phenols. Interfering absorption by mercaptans and hydroperoxides were found to produce little effect. The deviation is seldom greater than 1 per cent of the phenol content.

(c) TAR ACIDS: Harrison²⁶ has used the Spekker photoelectric absorptiometer in a procedure for the determination of tar acids in coal distillation products. The method he describes involves the use of the well-known phosphomolybdic acid reagent in the absorption measurement of phenols in tar acids. Phenols reduce this reagent when saturated sodium carbonate solution is added to solutions containing phenols. A green colour first appears, and the colour deepens to a dark blue of maxi-

mum depth after standing for 30 minutes. Thereafter, the colour fades gradually. With 4-cm. cells, Ilford No. 608 red filters, and a setting of water-water of 1.0, plotting drum readings against concentration gave straight lines for phenol, *o*-, *m*- and *p*-cresol, and *m*-4-xyleneol, the slopes of the lines varying. The absorption of mixtures is the sum of those of the components. Analysis of effluent samples showed that other constituents did not interfere. The author, in a later paper²⁷, introduced a mercury vapour light source for the absorptiometric determination of phenol, to reduce the interference from cresols and to improve the accuracy of the method. The relationship between absorption and weight of phenol is linear, provided that a constant volume of test solution is employed. Absorption measurement is simple, and more accurate and convenient in operation than colorimetric comparisons.

A freezing-point method for the determination of phenols in mixtures of tar acids gives satisfactory results²⁸. Equal weights of the three cresols produce equal lowering of the temperature at which phenol hydrate separates from mixtures of the acids with water. For the determination, the tar acids are freed from all but 2-3 per cent of neutral hydrocarbons and pyridine bases. Xylenols or higher homologues are removed by fractionation with a good column. The mixture is then brought to above 55 per cent phenol by adding a known weight of pure phenol. Ten per cent of its weight of water is added to the mixture and the freezing-point determined by noting to 0.1°C. the temperature at which the cloud of fine crystals disappears. The percentage of phenol in the anhydrous mixture and the freezing-point of the mixture when 10 per cent of water is added are known, and the unknown value can be readily calculated. The method can be used for crude carboic acid, when the latter is freed from water and higher homologues by fractionation and salting out. It is rapid, and not greatly influenced by small traces of moisture in the sample.

Gas & Coke Plant Wastes

(d) WASTE LIQUORS: Bach and Uthe²⁹ determined phenol in gas liquors and in waste water from coke plants by the classical gravimetric method of Köppeschaar. 100 ml. of phenol-containing liquid is evaporated to small volume with 5 ml. of

sulphuric acid, cooled, and the residue extracted with 100 ml. of water. The extract is then distilled until only 10 ml. remains. The residue is then distilled until only 10 ml. remains. The residue is then distilled until only 10 ml. remains.

With a 100 ml. flask, heat 50 ml. of 30 per cent sodium hydroxide to 60°C., and read the volume. Add 100 ml. of benzene and the distillate from 100 ml. of the tar oil. Shake thoroughly, again place in 60°C. water-bath and read the volume of the alkali.

The increase minus the water content of the oil is the percentage of phenol. A good separation of oil and alkali takes 1 hour. The method gave the highest results of the four used on samples of lignite tar and other tar oils.

Phenols in petrols and cresylic acids can be determined by a u.v. spectrophotometric technique, using the absorption band at 290 m μ . The phenols are extracted with an aqueous solution of sodium hydroxide. An empirical specific extinction coefficient of 24 was used after investigation of some pure phenols. Interfering absorption by mercaptans and hydroperoxides were found to produce little effect. The deviation is seldom greater than 1 per cent of the phenol content.

sulphuric acid, and the solution diluted to 100 ml. with water and distilled. The distillate is filtered, and heated under reflux until all the hydrogen sulphide present is evolved. 1 ml. of N barium chloride solution is added, followed by sodium hydroxide solution until alkaline. The liquid is filtered free from barium sulphate and carbonate, and an aliquot taken representing 10 ml. of the sample. The Koppeschaar method, in which the phenol is weighed as tribromophenol bromide, is then applied.

Williams¹⁰⁰ has described an analytical procedure suitable for determining phenols in ammonia liquor and ammonia still waste. The phenols are precipitated with bromine and the residual bromine determined. Ammonia, cyanides and sulphides must be removed before analysis, as these react with bromine.

The rapid titrimetric method of Munz¹⁰¹ has also proved satisfactory for the determination of phenol in these liquors. 25 ml. of the filtered liquor is measured into a 250 ml. distillation flask. Add a saturated solution of copper sulphate in ammonia (0.88) in excess of precipitation and then 5 ml. of 40 per cent sodium hydroxide solution. Boil until ammonia vapours are no longer evident, connect the flask to a condenser and pass carbon dioxide through the solution at a rate of 2-3 bubbles per second. The neutralisation should be complete in about 5 minutes. Distil to dryness with a slow stream of carbon dioxide. Rinse the condenser into the 250 ml. receiving volumetric flask and make up the distillate to volume. For titration, 25 or 50 ml. of this is used in the case of ammonia liquors, but with waste liquors the whole may be necessary. To this in a stoppered flask, add 25 ml. of 0.1N bromate-bromide solution and then 15 ml. of 15 per cent sulphuric acid. After 30 minutes, add 10 ml. of 10 per cent potassium iodide, stand for 15 minutes and titrate the excess with 0.1N sodium thiosulphate solution.

Conductometric Titration

The conductometric titration of phenols in the waste waters from fuel pyrolysis has been reported by Lapshin and Nikolaev¹⁰². To determine the amount of acid required for the neutralisation, titrate conductometrically 25-100 ml. of the waste waters with N sulphuric acid. To another 100-200 ml. of sample, add 5 per cent excess sulphuric acid

and 10 per cent copper sulphate solution (to precipitate hydrogen sulphide), reflux the solution for 30 minutes to expel any volatile matter, and filter. Distil the filtrate with steam into 6-10 fractions of 100-200 ml., reflux each fraction for 30 minutes, make up to a 250 ml. volume and proceed with the conductometric titrations of the aliquot parts (10-25 ml.) of each fraction.

Two titrimetric methods for the determination of phenol in waste liquors are due to Dehe¹⁰³. The first is iodometric:—To 200 ml. of sample add 10 ml. of 9N sulphuric acid, and shake for 3-5 hours. Filter, acidify the filtrate with dilute sulphuric acid, and distil with steam until the distillate no longer gives a positive test for phenol with Millon's reagent. Make the distillate alkaline, evaporate to dryness, transfer the residue with water to a 500 ml. volumetric flask and fill to the mark. Dilute 10 ml. of this solution with water, warm to 50°-60°C., add 15 ml. of 0.1N iodine solution, cool, acidify with dilute sulphuric acid, and back-titrate with 0.1N sodium thiosulphate.

Acidimetric Method

The second method is acidimetric:—Mix 50-60 ml. of the sample with a small amount of baryta, shake, and allow to stand for a few minutes. Filter, mix the filtrate with a little dilute sulphuric acid, filter off the barium sulphate, and wash the filter 2-3 times with small volumes of water. Add exactly 0.2 ml. of a 1 per cent alizarin yellow solution, and adjust the pH to 11.04 using a suitable buffer solution. Then add phenolphthalein indicator and titrate with 0.1N sulphuric acid to pH 8.4, again using a buffer. From the number of ml. of 0.1N acid used, the correction for pure water is subtracted. Under normal conditions the ml. of 0.1N acid used, multiplied by 2.09, gives the number of g. of phenol per litre of waste liquor.

Rose and Sperr¹⁰⁴ have developed the following method for the rapid control determination of phenols in ammonia still waste: To 10 ml. of the filtered sample, add dilute bromine water (saturated bromine water + 4 times its volume of water) dropwise until a slight permanent yellowish tint is observed. If an immediate and heavy white precipitate forms, phenols are present in a concentration greater than 1 in 10,000. If no crystalline precipitate appears after 5 minutes, the phenol content is below 1 in

50,000. The amount of phenols is determined by diluting a known amount of the liquor with water until it gives a test from a slight milkiness to a faint crystalline precipitate, and comparing it with standard samples. It will be found very simple to select the proper standard.

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Henry Balfour & Co.

Scots Engineering Firm Expands

A STATEMENT by the directors of Henry Balfour & Co., Ltd., engineers and iron-founders, Durie Foundry, Leven, Fife, concerning the affairs of their company was issued last week, applications having been made to the Committees of the Stock Exchanges of Edinburgh and Glasgow for permission to deal in and a quotation for the outstanding 5 per cent Convertible Unsecured Loan stock and the issued Ordinary shares of the company.

The company, founded in 1810 by Mr. Henry Balfour, is one of the oldest engineering businesses in Scotland, and the acquisition of interests in various companies from time to time has resulted in considerable expansion of much specialised work. It is claimed that the company is now in a leading

position in the chemical and allied engineering industry of Great Britain.

The entire production is concentrated at Durie Foundry, where the output consists of specialised plants for scientific undertaking throughout the world and many of the products of the company and of their subsidiaries are protected by patent. Among the industries served are gas, heavy chemical, medicinal, soap, glycerine, paper, milk, dyest, brewing and distilling, plastics, artificial rubber, and animal by-products.

Productivity has multiplied many times since 1929 and is still increasing and the company contributes directly and indirectly, and very substantially, to the U.K. export trade.

The directors are satisfied that, despite shortages of raw materials, trading results for the year to 31 October next will be sufficient to justify the maintenance of the 22½ per cent dividend on the Ordinary shares. An interim of 10 per cent payable on 31 October has already been decided upon.

The net combined assets of the group before deducting the Unsecured Loan stock, are given as £521,636, which is sufficient to cover the amount of loan stock outstanding, namely, £178,200, nearly three times. After deducting all prior charges, including £6,000 in £1, 5 per cent, Preference shares and £19,000 in £1, 6 per cent, 'A' Preference shares, a figure of £318,436 is equal to about 24s. 6d. per 5s. Ordinary share issued.

On the basis of profits at £133,244 for the year to October 31 last and after providing for all taxation and the redemption of Unsecured Loan stock and for Preference dividends, the balance would be sufficient to cover more than five times the Ordinary dividend of 22½ per cent on shares at present issued.

Z.D.A. Abstracts

OUTPUT of scientific material has reached such large proportions that it is almost impossible to keep up to date with all new developments. *Z.D.A. Abstracts* published by the Zinc Development Association provide a valuable review of recent technical literature on the uses of zinc and its products, with other material of interest. Latest additions are number 3, 4 and 5 of Volume 10, containing abstracts numbers 161-222, 223-309, and 310-374 respectively.

Newer Inorganic Pigments

A Survey of German Research

INVESTIGATION of inorganic pigments on an extensive scale has formed an important part of the research programme of Farbwerke Hoechst, of Frankfurt/Main. Work of the company during the last two decades on blue pigments suitable for cement and on other pigments and anti-rust preparations has recently been described by Dr. E. Korinth (*Agnew. Chem.*, 1952, 64 (9-10), pp. 265-269).

This problem of finding a blue pigment fast to cement is by no means new. A prize for an essay on this subject was indeed won in a French competition (Soc. d'Encouragement) by J. B. Guimet as long ago as 1828.

Discussing first Rinman's green and similar compounds, the author points out that this green pigment is obtained by heating those compounds of zinc and cobalt that decompose to form oxides. While zinc oxide crystallises in the wurtzite structure, cobalt (II)-oxide crystallises in the face-centred cubic system of the sodium chloride type.

According to Natta and Passerini dark green products are obtained by heating material containing considerable amounts of trivalent Co to 800-1,000°C., in the presence of air. Analysis by X-ray shows that this mixture consists of cubic zinc cobaltic and hexagonal crystals practically identical with those of ZnO, which has the wurtzite structure. In the absence of oxygen all the Co is present as divalent.

Solid Hexagonal Solutions

Such compounds consist of solid hexagonal solutions of CoO in the ZnO lattice (according to Hedvall, ZnO and Co zincate). In this condition the CoO lattice, which otherwise requires a temperature above 350°C., is stabilised. The same holds also for the otherwise chemically unstable divalent phase. Crystals with more than 70 per cent CoO, on the other hand, consist of solid cubic solutions of ZnO in the CoO lattice. Thus an isodimorphous mixed crystal system is present. The hexagonal product is a bright green; in the presence of trivalent Co the colour is darker.

Corresponding in chemical structure to Rinman's green is magnesia red consisting of mixed crystals of regular (normal) MgO and

CoO. Comparison of the lattice constants of CoO with those of NiO, FeO, MnO, CdO, MgO and ZnO support the conclusion that it must be possible—with iron (II)—oxide or manganese (II)-oxide, together with ZnO—to produce pigments analogous to Rinman's green in constitution. This is strengthened by the fact that coloured zinc ores are known in nature, but they have not so far been achieved in paint technology.

Since, for example, FeO and MnO are oxidised much more easily than CoO, compounds of the higher oxides of these metals with ZnO are obtained on heating in air; but the light to dark brown colours of these oxides are not particularly impressive. Conditions must be sought therefore under which MnO or FeO may be produced. As shown for the system Fe-C-O (Gmelin-Kraut 8th ed., system 59) the metal oxide phase is only in equilibrium if, besides C or H₂, water vapour or CO₂ are present, the temperature range being 450° to 1,300°C.

Confirmed By Tests

These ideas were confirmed by actual test. See Korinth's German Pat. No. 589,783, where MnO is used instead of CoO for production of yellow to bluish-red pigments.

Of the numerous ternary systems giving surprising colour effects, three may be named by way of example. They were studied by means of triangular co-ordinates with molar per cent variations in composition. Interesting blue, violet, and black tones are to be found in the ternary system ZnO-CoO-SiO₂; and in the system NiO-ZnO-TiO₂ an extraordinary pure or whole colour laurel green is obtained due largely to the presence of zinc. In the CoO-Al₂O₃-TiO₂ system a whole range of blue, bluish-green to black colour shades are obtained. Black pigments are obtained in the Zn,Fe,Ti oxide system, and so on for several others including boron or tungsten oxides yielding beautiful violet, blue and other colours.

Manganese blue is probably a class by itself. Cobalt has been mainly used for blue pigments, but is expensive. And the chief difficulty with manganese blues for some time had been their relative instability, for example, when prepared from Ba and Mn

compounds. Consideration of Grimm's mixed crystals with supplementary roasting thereof, or the introduction of Mn into the barium sulphate lattice by heating, led to the desired result, as described in several of Korinth's German and foreign patents.

The Grimm mixed crystals are prepared and heated to 700°C. in the presence of barium nitrate, yielding a mixed crystal of barium sulphate and basic barium manganate (German pat. 651,683). The comminuted roast material is further treated in the presence of a reducing agent, yielding sulphate ions; and the manganate ions are replaced by barium sulphate. The blue pigment in suspension treated with lead chromate/lead sulphate and the mixed crystal becomes covered with a yellow coating and is converted to a bluish green pigment; but the stability of the yellow coating is rather poor, for example, against rubbing or friction.

Finally, Dr. Korinth deals with his firm's anti-corrosive research extending over many years; with special reference to the development and use of lead cyanamide and the attempt to reduce lead consumption as much as possible in view of a probable increasing world shortage of this metal.

Lead or barium ferrites proved to be a promising line (German pats. 651,684 and 684,659), yielding pigments of high anti-corrosive efficiency. A general description of lead cyanamide in this connection is given, including its use in linseed oil varnishes, soap formation and so on, with illustrative micro-photographs.

German Symposium on Fats

FURTHER particulars of the fourth post-war annual general meeting and symposium of the Deutsche Gesellschaft für Fettwissenschaft (DGF) to be held in Düsseldorf (THE CHEMICAL AGE, 67, 139), are now available.

The correct dates of the symposium are from Sunday, 5 to Thursday 9 October, and not from 6-10 October as originally announced.

Of some 60 papers already received, 16 are devoted to general and physical chemistry of fats, and analysis. The next largest classification is machinery and equipment (10), the remainder being divided among: oilseeds and edible fats (seven); miscellaneous (six); biology, etc., and cos-

metics and pharmacy (five each); soaps and detergents and paints and varnishes (four each), waxes (three).

Most of the speakers, naturally, come from Germany, but visiting lecturers will include three from Great Britain and the Commonwealth; three from Austria; two each from Italy and Switzerland, and one each from Belgium, the Netherlands, Spain and Sweden.

Dr. J. Lovern, D.S.I.R., Torry Research Station, Aberdeen, will give a paper on 'The Origin of the Peculiarity of Fatness in Fish'. Dr. L. Ivanovsky, London, will speak on 'Latest Results of Solvent-Retention'; and Dr. J. B. Shorland, Wellington, New Zealand will describe 'New Investigations in the Field of Fats, especially in regard to the work of the New Zealand Institute for Fat Research.'

A number of social events for members attending the meeting have been arranged for the evenings, and the last day will be devoted to a series of works days.

IN THE EDITOR'S POST

A Further Protest

SIR,—We regret having to give you the news of the stoppage at the end of this week of all further development and production work at our barytes mines at Closehouse and Lunehead, Middleton-in-Teesdale, in the North Eastern Development Area, and to offer the property for sale forthwith.

The Closehouse Mine in particular has operated very successfully since 1939 and has important ore reserves and good prospects.

The stoppage has become necessary to protect our interests as independent producers of barytes and as a further protest against the persistent practice of Government departments treating non-statutory voluntary bodies as the sole channel of communication with the industry to our detriment as non-members. Recently a letter of ours to the Board of Trade was actually passed to the Association of British Chemical Manufacturers for reply to us!

Yours faithfully,

ATHOLE G. ALLEN.

Governing Director,

Athole G. Allen (Stockton), Ltd.

Metallurgical Coke

A Prosperous Market for Petroleum Refineries

(by an American Correspondent)

ONE of the neglected products of petroleum refining has been metallurgical coke. As a result, the market value of this material has risen until today it commands some \$21.00 per ton in the United States. This compares with a price of \$4.50-6.00 per ton being quoted today for fuel grade petroleum coke.*

The market for metallurgical-grade petroleum coke has been on the rise for many years now. It has been estimated that the total 1950 production of petroleum coke was some 3,420,000 tons or 1.2 per cent of crude oil run to stills. Of this, about 48 per cent served as fuel in either domestic or industrial installations. Fully 20 per cent, however, goes into the aluminium industry and another 10 per cent is utilised in the production of mild and alloy steels.

In 1950, the aluminium industry alone consumed some 900,000 tons of low-ash coke. Since a product of suitable quality was not forthcoming from the petroleum industry in adequate amounts, a fair share of this market was covered by coke derived from coal distillation pitch. Actually, the petroleum industry finds itself in a more ideal position than the coal carbonisation industry to satisfy the demand for metallurgical coke. Yet it has been only during the last few years that serious efforts have been made to satisfy the rapidly growing demand for this important product by the coking of heavy oils.

Specifications Stringent

Specifications for metallurgical coke are stringent and limit the suitable raw materials severely. The volatile matter must be in the range 4.5-8 per cent, sulphur content must not exceed 0.5 per cent while the ash must be below 0.75 per cent. Greater leeway is allowed in the demands made on petroleum coke for aluminium electrode production which, again in 1950, consumed 7 per cent of the total output of petroleum coke. Here, volatiles must be below 12 per cent while the sulphur content must be less than 2 per cent and the ash content must

not exceed 1 per cent. In addition, it is desired that the metallurgical coke be of uniform quality and high specific gravity.

In today's petroleum industry, the bulk of coke is produced by delayed coking operations, i.e., by a process in which heavy fuel oil or gas oil is heated to temperatures above 900°F. and is then permitted to dissociate into coke and light overhead by 'soaking' in a coke drum. This operation is continued until the drum is filled with coke, after which it is taken off stream and the coke is broken out by mechanical or hydraulic means.

High Volatile Content

Coke produced in this fashion tends to have a volatile content in the range of 8-18 per cent, i.e., higher than the specifications for most metallurgical coke will permit. The sulphur- and ash content, of course, depend on the nature of the crude as well as on additions made in the course of refining. Coke hardness depends on operating conditions employed. Thus, it is possible to raise the degree of hardness (and, incidentally, reduce volatile content) by operating at higher temperatures, employing longer soaking time, or using feedstock of low hydrogen content (i.e., heavy fuel oils). Here, however, a limit is set by the high operating temperatures. Very heavy stocks, such as asphalts, pitches and reduced tars cannot be satisfactorily raised to the desired temperatures in fired tubular heaters due to their tendency to coke rapidly above 900°F. and cause fouling of the pipe coil.

By its ability to yield, simultaneously, coke and large amounts of adequate catalytic cracking feedstock, the delayed coking process has become an important and integral part of many refineries today. As far as coke characteristics are concerned, some of the limitations encountered have been enumerated above. In addition, the cyclical nature of the process results in the formation of relatively non-uniform coke insofar as hardness, specific gravity, and volatile matter are concerned. The bottom portion of coke in the soaking drum, having been exposed to coking conditions for a more extended period, will fulfil the specifications for metal-

* Blast furnace coke, as used by British steel makers, costs approximately £4 16s. 6d. a ton.

lurgical coke more commonly than the coke formed during the later stages of the cycle and deposited in the upper part of the chamber.

Other methods are being used on a smaller scale for the production of petroleum coke. Included, merely for the sake of greater completeness, is the simultaneous coking and thermal cracking operation employed for the production of maximum yields of coke and gasoline. In this scheme, gas oil is heated to 1,100°F. at 500 p.s.i.g. Immediately upon issuing from the pipe coil, it is quenched with residuum or heavy fuel oil to a temperature of 800-875°F., at which the mixture enters a coke chamber. This operation overcomes some of the limitations exhibited by delayed coking in regard to the use of heavy oils. However, this is done at the expense of a fair portion of valuable gas oil. This operation, too, is cyclical.

Heavy Oil Coking

Still another method employed only on a limited scale is the coking of heavy oil in externally heated refractory ovens of the 'Knowles' type. Operating temperature is up to 1,500°F. and a product of high quality and uniformity is obtained. However, both investment and operating costs are high in this process.

Continuous contact coking, a recent development by the Lummus Co., represents an ingenious contribution in this field toward the production of high quality petroleum coke with simultaneous formation of catalytic cracker feedstock in high yield. This process is an outgrowth of the thermoform catalytic cracking (TCC) method and its validity has been established in semi-commercial operation. Now under construction is a commercial unit, designed for this process for Continental Oil Company's Poncas City refinery.

In this process, the hydrocarbon feedstock is preheated to 750-850°F. in a conventional tubular preheater. In this temperature range the viscosity of even the heaviest residual stock is low and the material is characterised by a high degree of pumpability. At the same time, conditions are not yet so drastic that cracking and fouling will occur within the tubes.

The hot oil is brought into immediate contact with a large quantity of hot circulating coke in the form of rounded lumps in the $\frac{1}{4}$ in.- $\frac{3}{4}$ in. size range. This carrier coke has

been formed in the process itself. It is found that the hydrocarbon oil wets the solid particles quite uniformly in very thin layers.

From the point of contact, the hot, wetted coke is permitted to travel by gravity through the reactor zone in which it is given a soaking time of 15-40 minutes. From here, the freely moving particles pass through a sealing zone into a reheater where they are once again raised to proper re-circulation temperature by exposure to radiative and some convective heating. From here, the hot coke is returned to the point of contact with the hydrocarbon stock by means of mechanical elevators or vapour lift systems which have long since been established in refinery practice.

A portion of the coke particles is continually removed from the system as product. The removal generally takes place via a sizing unit so that the granules below $\frac{1}{4}$ in. size may be returned to the process. By the installation of proper sizing rolls, the amount of oversize in the reactor zone may be limited.

The vapours leaving the reactor are quenched by means of a stream of heavy hydrocarbons. Because of the large heating surfaces involved, distillation of the volatile components is almost instantaneous and cracking (or at least secondary cracking) is at a minimum. As a result, the formation of gasoline and hydrocarbon gas is considerably below that encountered in delayed coking. While these light fractions, formed by cracking of heavier oils, are relatively unsaturated, the gas oil formed in the process shows little effect of thermal degradation. It is quite saturated and is nearly as suitable for catalytic and thermal cracking operations as the virgin gas oil itself.

Quality Remarkably High

The quality of the resultant coke is remarkably high and uniform due to its formation in exceedingly thin layers. It is reported to have 1.5-5.0 per cent volatile matter and a bulk density of 58-60 lb. per cu. ft. The sulphur content as well as the ash will depend on the crude charge. The coke is relatively low in porosity and is very high in mechanical strength (individual particles have been reported with a crushing strength of 600 p.s.i.). Thus, this grade of petroleum coke will satisfy most metallurgical requirements provided that the sulphur- and ash content are low enough. In

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It is addition, its low content in volatile matter renders it an ideal powdered fuel after crushing.

Thermal economy can be achieved by calcining the coke immediately at its point of issue from the reactor, if electrode coke is the desired product.

From the point of view of overall economy, the high quality of the products and the excellent oil yields are of paramount importance. A comparison of performance obtained in continuous contact and in delayed coking, as reported by Schutte and Offutt, is shown in Table 1:—

TABLE I
Comparison of Contact and Delayed Coking Yields
Recycling to Ultimate Yields

Operation	Contact Coking (Mid-Continent Crude)	Delayed Coking Reduced Crude
<i>Charge</i>		
<i>Reduced Crude Inspection</i>		
Per cent on crude ..	26.5	26.5
Gravity, °API ..	20.6	20.6
Carbon residue ..	8.2	8.2
<i>Product Yields, per cent on</i>		
<i>Coker Charge—</i>		
Gas, wt. per cent ..	6.0	7.0
10 R.V.P. gasoline, vol. per cent ..	14.9	21.6
Gas oil, vol. per cent ..	75.5	66.5
Coke, wt. per cent ..	11.5	14.0
Gas oil endpoint, °F. at 760 mm. ..	830	830

This process is expected to widen the range of raw materials which can be processed by continuous thermal refining methods. An example is Canada's Alberta Oil Sands which are handled only with difficulty in more conventional processes because of their high coking tendency and consequent inclination to foul heater tubes. In continuous contact coking, this difficulty is neatly side-stepped.

issue on the same terms at the option of the company up to 31 December, 1954, on payment of a commitment fee at the rate of one per cent per annum up to the time of issue. It is not intended to seek a quotation on any Stock Exchange.

Annual Sinking Fund

An annual non-cumulative sinking fund for the redemption of the stock at par is to commence in 1963 equal to $2\frac{1}{2}$ per cent or (at the company's option in any year) five per cent of the stock outstanding on 1 July, 1962. The company has the option to redeem all or any part of the stock after 31 December, 1953, at varying premiums commencing with 110 per cent up to 30 June, 1957, at $107\frac{1}{2}$ per cent after that date and up to 30 June, 1960, and descending thereafter by one per cent for every three years down to 30 June, 1980, and thereafter at par. Any of the stock not previously redeemed is repayable at par on 1 July, 1982.

Stock will be repayable at the option redemption price for the time current in the usual events (such as winding-up or breach by the company of the conditions of the deed constituting the stock); and also if the company declares or pays any dividend at a time when the net tangible assets (as defined in the deed) of the company and its subsidiaries are less than twice the amount of the funded debt (as defined in the deed) of the company and its subsidiaries and the holders of not less than 60 per cent of the stock call the stock for repayment.

Although the stock is unsecured the company and its subsidiaries may not without the sanction of the holders of the stock:

(a) with certain exceptions (such as pledges of current assets to secure current liabilities) create mortgages or charges without similarly securing the stock; or

(b) create or assume funded debt (as defined in the deed) unless (i) net tangible assets of the company and its subsidiaries are equal to 200 per cent of funded debt and any indebtedness of third parties guaranteed by the company or any of its subsidiaries, and (ii) the average consolidated earnings of the company and its subsidiaries for the three preceding years equal $2\frac{1}{2}$ times the annual interest requirements of all consolidated funded debt of the company and its subsidiaries and any interest payable by third parties and guaranteed by the company or any of its subsidiaries.

Chemical Financial Loan

Monsanto's £3,500,000 Agreement

THE directors of Monsanto Chemicals Ltd., announce, in connection with the financing of its plans (foreshadowed in the chairman's statement accompanying the accounts for 1951), that it has entered into an agreement with the London offices of the Canada Life Assurance Company, Sun Life Assurance Company of Canada, and Confederation Life Association of Canada, whereby those companies have agreed to subscribe privately in certain proportions for cash at par for £3,500,000 five per cent Unsecured Loan stock, 1982, the first £1,500,000 of which has been issued to them. The balance £2,000,000 is available in stages for

Fighting the Locust in Iraq

Aerial Spraying with New Insecticide

ONE of the worst locust plagues in living memory has just been overcome in Iraq by prompt action by local government authorities, including aerial spraying of the modern petroleum-derived insecticide—'Aldrin', of which Shell has the sole manufacturing rights.

Originating in Ethiopia and Somalia at the beginning of this year, the locust plague swarmed across the Red Sea and, by the end of February, had spread over Southern Iraq and Iran. The agricultural economy of Iraq was in immediate danger because most of the land between the Tigris and the Euphrates is under crops, including cereals, maize and vegetables.

Control operations were started at once. They were of two kinds. Ground control was initiated by the Iraqi local governments who called on all available manpower for the application of poison bait. In areas where it would have been very difficult, if not impossible, to control with the bait method, arrangements were made for two Piper Cub aircraft to spray 'Aldrin' 60 per cent emulsible concentrate from the air.

The aerial spraying programme proved to be 100 per cent successful. In the regions lying alongside the banks of the River Tigris, for example, where aircraft spraying was the only practical method of control because of the dense vegetation, heavily infested with hoppers, the locusts were reported to be completely eradicated after two days' work.

'Aldrin' not only eradicated third and fourth stage hoppers within 48 hours but was found to be still effective seven days later against a new invasion.

Giant U.S.A. Merger

ONE of the world's leading manufacturers of pharmaceuticals, E. R. Squibb & Sons, is to be merged into the Mathieson Chemical Corporation of Baltimore, Maryland. It will retain the name and will continue the policies and standards for which it has been known so long, and it will be operated as a separate division of Mathieson.

Of the two companies, E. R. Squibb & Sons is the older, having been started in 1858 whereas Mathieson was founded in 1892. The

former occupies a leading position in the field of antibiotics, vitamins, anaesthetics, sulphonamides, etc., as well as a broad line of medical household products. The Mathieson Chemical Corporation is one of the leading American chemical companies and in recent years has undergone a major expansion in basic chemicals, agricultural chemicals and petrochemicals. It now operates 20 plants in 10 states.

At present the aggregate sales of the two companies are running at the annual rate of over \$200,000,000 (approximately £71,428,571) and their combined gross assets are about \$335,000,000 (£119,642,857), and working capital exceeds \$85,000,000 (£30,357,142).

In a letter to shareholders the president of the Mathieson Chemical Corporation, Mr. Thomas S. Nichols, president and chairman, has said that the merger will strengthen both companies, will provide greater diversification and will offer increased opportunities for expanded research and development.

Oil Cartel Alleged

IN Washington on 7 August President Truman is reported to have said that he was considering making public the report prepared by the Federal Trade Commission on the operations on an international oil cartel. At the same time 21 oil companies were said to have had subpoenas served on them, at their New York offices requesting them to produce data on their international transactions from 1 January, 1928, up to 15 July, 1952.

The subpoenas were issued in connection with an investigation by the United States Department of Justice into the alleged cartel arrangements and are returnable on 3 September before a grand jury in the criminal division of a district court in Washington.

The companies concerned includes: The Standard Oil Co. (New Jersey), Anglo-Iranian Oil, Iraq Petroleum, Socony-Vacuum Oil, Standard Oil Company of California, Shell Trading and Transport, Royal Dutch Petroleum, Gulf Oil Corporation, Atlantic Refining, The Texas Company, Pan American Petroleum and Transport, Standard Oil (Indiana), Superior Oil Company of California, American Independent Oil, Kuwait Petroleum, Arabia-American Oil, Standard-Vacuum Oil.

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The Chemist's Bookshelf

CHEMICAL TRADE NAMES AND COMMERCIAL SYNONYMS. By Williams Haynes. Macmillan & Co., London; D. Van Nostrand Company Inc., New York. 1951. Pp. iv + 279. Price: 41s., \$5.50.

This very full reference book to all the trade names and chemical synonyms of America has both its strengths and its weaknesses. Fortunately, the part which will be of most use to people on this side of the Atlantic is excellent.

Mr. Haynes has classified all the trade names in use up to January, 1950, in the U.S.A., compiling this mammoth list from the answers to nearly 500 letters of inquiry received from the actual manufacturers themselves. This will be of particular value in this country and on the continent, because so often it is very difficult, without recourse to some outside agency, to trace the meaning or source of some proprietary American trade name. Another asset is the inclusion after each trade name of the registered owner, an incalculable advantage when one is faced with some obscure name which nobody owns or even recognises, more especially so when the firms likely to know are not accessible by telephone. Registered trade marks have been taken from the *Patent Gazette*, and new products and 'specialities' from various authoritative journals. The book will appeal greatly on the strength of this portion alone.

If Mr. Haynes does less well on the chemical synonym side of his book, this is not to decry its usefulness in its other capacity. Trade names are fairly simple things to include or omit. Chemical names by comparison are labyrinths of complexity. The compiler must decide how many to put in and how many to leave out, how he will define those he does put in, and for what kind of public he will do it.

This book's weakness is its inconsistency and inadequacy in this field. Mr. Haynes says in the beginning that he bears in mind 'the layman who will consult these pages', but in places he does this a little too well,

at the expense of space which could be better employed. Drawing the line is always difficult, but surely the entry 'Crude oil: petroleum' is an elementary truth that could with advantage have been left out, since those in ignorance of it are scarcely likely to possess this book at all. Furthermore, the inclusion of 'Appeal (Chiris): perfume base', 'Paradise' and 'Tempress', made the reviewer immediately look up under 'My Sin' and 'Tabu' to see how far the author had committed himself. Oddly, natural ambergris itself could not be found, although several synthetic varieties were mentioned.

On the other hand, Mr. Haynes makes some rather inexplicable omissions. If he is going to include for the layman an explanation of the terms 'ethanol' as being 'ethyl alcohol', and 'chloral' as being 'trichloroacetaldehyde', he must in all conscience include chemical designations for the random examples of cresol, resorcinol, picric acid, thiamine, antipyrine, etc., many of which are mentioned but not defined, although substances such as pyrogallol, anisole, anabasine, amidopyrine, pinene and dekaline are.

A more serious omission from the point of view of the technical user, however, is the evidently complete exclusion of all ore names—neither cryolite, dolomite, fluorspar, olivine, not even pyrites or anhydrite could be found. Moreover the descriptions set against many chemical terms are often misleading, if not actually incorrect. Thus 'Grain oil' (fusel oil) is described as 'amyl alcohol'. It is not amyl alcohol—60 per cent of it—and never has been, but a mixture. 'Leuco. dye' is explained simply as 'vat dye, q.v.' Under 'Vat dye' the author explains the use of the soluble, colourless form and the insoluble form, but without saying which is the leuco form. Several more examples could be given, although the cross-indexing is generally quite good.

There are also fairly frequent printing errors in the book, on occasion completely destroying the sense of words. Thus pinane is defined as '2,4-bichloheptane'. Pinene goes

one better and is called '2,4-bicycloheptane.'

By and large, this book must be regarded mainly as an index to the trade names of America. As such, it will be extremely useful.—J.C.S.

STRUCTURAL CHEMISTRY OF INORGANIC COMPOUNDS. Volume II. By W. Hückel. Translated by L. H. Long. Elsevier Publishing Co., Ltd. Distributors: Cleaver-Hume Press, London. 1951. Pp. x + 654. Figs. 120. 90s.

This volume, which represents the third and final book of the original work, completes the translation of which the first volume has already been reviewed (*THE CHEMICAL AGE*, 1951, 64, 967). The sub-title of the volume is 'Structure and Constitution,' and there are six main sections, as follows: Volatility of Inorganic Molecules; Crystal Chemistry; Silicates and Glasses; Metallic Substances and Alloys; The Chemical Reaction in Inorganic Chemistry; Lines of Research in Chemical Science.

It has already been made clear in the review of the first volume that the actual process of translation leaves something to be desired, and the labour of reading the book is perhaps as much due to the language in which the concepts are presented as to the inherent difficulty of these concepts. This is particularly unfortunate since the order of presentation of the material is frequently unusual, so that to be properly and easily appreciated it would demand the clearest possible expression.

The approach of the author throughout lays very heavy stress on the structural consideration of inorganic chemistry, occasionally resulting in a certain lack of descriptive material. However, the fresh treatment is undoubtedly salutary, and should lead to fresh thoughts on the part of the reader about many sections of inorganic knowledge. Occasionally the author is forced by his premises into quite frankly jettisoning the treatment which he has used for one group of compounds, as being inappropriate for another group. Thus, in the valuable discussion on volatility he is forced to admit that the rules postulated in considering the holidos do not serve when considering the elements, and a completely fresh approach must be made to the latter group. This is an indication both of the lack of fundamental knowledge which

still exists and of the magnitude of the task which the author set himself.

In addition to the section on volatility, those on morphotropy and on reactions in the solid state may perhaps be picked out either as treating an older subject in a novel way, or as presenting a collection of material not previously collated satisfactorily. However, there are many other parts of the book which could, with almost equal justification, be chosen for special mention.

That the author is some considerable way from achieving his aim—to present for the inorganic chemist a combined structural and constitutional theory such as that which has allowed so much systematisation in the field of organic chemistry—is clear from the very short final section on lines of research. This is in many ways merely a restatement of what has been said at much greater length in the earlier chapters, and does not present the synthesis that one would expect from its title. It is doubtful if it adds much to the book.

However, by travelling hopefully much has been achieved. The reviewer has stressed that this is not an easy book to read. It is nevertheless well worthy of a place on the shelves of the inorganic chemist, and, indeed, cannot safely be omitted from any reasonably comprehensive collection on the subject. The vast amount of information contained can be estimated from the fact that the indexes—three in number, listing authors, subjects, and elements and compounds—occupy no less than 118 pages. The novel juxtaposition of topics in many cases, and the collation of hitherto isolated material, are stimulating.

The production of the book is excellent and calls for only one comment, arising from the somewhat unwieldy nature of this second volume. The fact that it is almost twice the size of the first volume causes one to speculate whether a more equitable partition, or, preferably, a division into three volumes rather than into two, might have been more acceptable.—C.L.W.

Zinc Prices Reduced

A reduction in the United Kingdom price of good ordinary brand zinc, by £12 to £118 a ton, with effect from 8 August, was recently announced by the Ministry of Supply. It was later announced that as from 13 August the price would be raised by £4 per ton.

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Change of Address

The London Sales Office of Britannia Batteries, Ltd., has just been transferred to new premises at 53 Victoria Street, London, S.W.1. Telephone number: Abbey 6168-9. Telegraphic address: Britanicus, Sowest, London.

Oils & Fat Prices Unchanged

No change will be made in the price of unrefined oils and fats and technical animal fats allocated to primary wholesalers and large trade users during the four-week period ending 6 September. Prices of refined oils and imported edible fats will also remain unaltered during the eight-week period ending 4 October, it is announced by the Minister of Food.

Glasgow Factory Taken Over

Marinite Limited, a joint company sponsored by Cape Asbestos Co., Ltd., and Johns-Manville Corporation, U.S.A. are to take over a 140,000 sq. ft. factory at Germiston, Glasgow. After alterations and installation of plant the company will manufacture pressed-asbestos board. Cape Asbestos will supply the raw materials from South Africa; and the new plant will go into full production in early 1953.

Technical & Scientific Register

The total number of persons enrolled on the Technical and Scientific Register at 16 June was 5,278. This included 4,158 registrants already in work but who desired a change of employment, and 1,120 registrants who were unemployed. During the five-week period, 13 May to 16 June, 770 vacancies were notified, 166 were filled, and 517 were cancelled or withdrawn.

Fatal Industrial Accidents

Only two deaths were reported in the chemicals, oils, soap, etc., category among the deaths from accidents in the course of employment in the United Kingdom in June. The total number recorded in *The Ministry of Labour Gazette* (Vol. 60, No. 7), was 125, compared with revised figures of 123 in the previous month and 104 in June, 1951. Metal extracting and refining accounted for three, metal conversion and founding, four, and other metal trades, two.

Big Capital Increase

Details have been released of an increase in the capital of Styrene Co-Polymers from £250,000 to £350,000. The new capital is in 37,500 6 per cent. Cumulative Convertible Preference and 62,500 £1 Ordinary shares. The company is owned jointly by Lewis Berger, Petrochemicals, and Pinchin Johnson.

Sir Charles Goodeve Rewarded

The Royal Commission on awards to inventors has recommended ex-gratia awards totalling £23,300 in connection with recent claims for devices designed to combat magnetic mines. Among those recommended for awards were Sir Charles Goodeve, Director of the British Iron and Steel Research Association, £7,500; Mr. W. F. B. Shaw, c/o Fuel Research Station, D.S.I.R. and Mr. H. Rowe, Directorate of Scientific Intelligence, £500 jointly.

Chemical Employment

A drop in the number of persons employed in the chemical and allied trades in Great Britain was recorded in the industrial analysis at the end of May. The total figures (in thousands) were 479.9 in May compared with 484.5 in April this year, and 489.3 at the end of 1951. Detailed distribution was: coke oven and by-product works, 17.9; chemicals and dyes, 217.4; pharmaceutical preparations, 44.6; explosives, etc., 49.7; paint and varnish, 39.6; soap, candles, polishes, etc., 48.0; mineral oil refining, 29.6; other oils, greases, glue, etc., 33.1.

New Plant Needed

A resolution to increase the capital of Quickfit and Quartz, Ltd. to £830,000 by the creation of redeemable cumulative preference shares was unanimously adopted at an extraordinary general meeting held in London on 6 August. Sir Graham Cunningham, chairman and managing director of the Triplex group of companies, said that the pressure for more cash to keep up-to-date by purchasing new plant came substantially from the three productive subsidiary companies, Quickfit and Quartz, Weldall and Assembly and Stern and Bell.

OVERSEAS

Italian Sulphuric Acid Production

Output of sulphuric acid in Italy in 1951 totalled 2,170,350 metric tons compared with 1,826,189 tons in 1950 and 1,855,868 tons in the previous year, according to data published by the Official Bulletin of the Central Institute of Statistics. Exports of sulphuric acid from Italy in 1951 showed an increase of 54 per cent over 1950, which year showed an increment of 10.87 per cent over 1949.

British Plastics in Denmark

The properties and qualities of laminated plastics and their ability to reduce costs of redecoration are becoming increasingly appreciated in Scandinavia, especially in shipbuilding. Denmark will be the scene of an export promotion scheme in the near future when an exhibition of Waverite Laminated Plastics will be held in the Byggencentrum (Building Centre), Copenhagen, from 22-31 August and then in the Aarhus Hall, Aarhus, from 5-7 September. Waverite materials are supplied in the United Kingdom by Waverite, Limited, a unit of Bakelite, Ltd. Overseas sales are handled by the export unit of Bakelite, Limited.

U.S.-Canadian Cobalt Deal

A long-term contract covering the sale of 1,000,000 lb. of cobalt has been signed by Silver Miller Mines with the U.S. Government. With the proceeds of the sale of cobalt concentrate this is expected to bring Canada \$15,000,000 in smelting and freight revenue. The Dominion's export regulations stipulate that only cobalt metal may be exported, so it is assumed that the concentrates will be smelted by the Delora smelting and Refining Company, Ontario.

Polyethylene Plant Planned

A new plant for the production of polyethylene and ethylene glycol is to be built outside Los Angeles, California, for the Carbide and Carbon Chemicals Company, a division of the Union Carbide and Carbon Corporation. The project will involve an eventual investment of some £36,857,143 and is being designed to produce from 50-60,000,000 pounds of polyethylene and from 5-10,000,000 gallons of ethylene glycol per year.

May Increase Output

It is reliably reported from New York that E. I. Dupont de Nemours and Co. are contemplating spending up to \$10,000,000 (£3,571,428) to expand production of nylon yarn at its Chattanooga, Tennessee, plant. It was stated that if the project under study for the manufacture of continuous filament nylon yarn was approved and authorised it would probably be completed some time in 1955.

World Tin Output

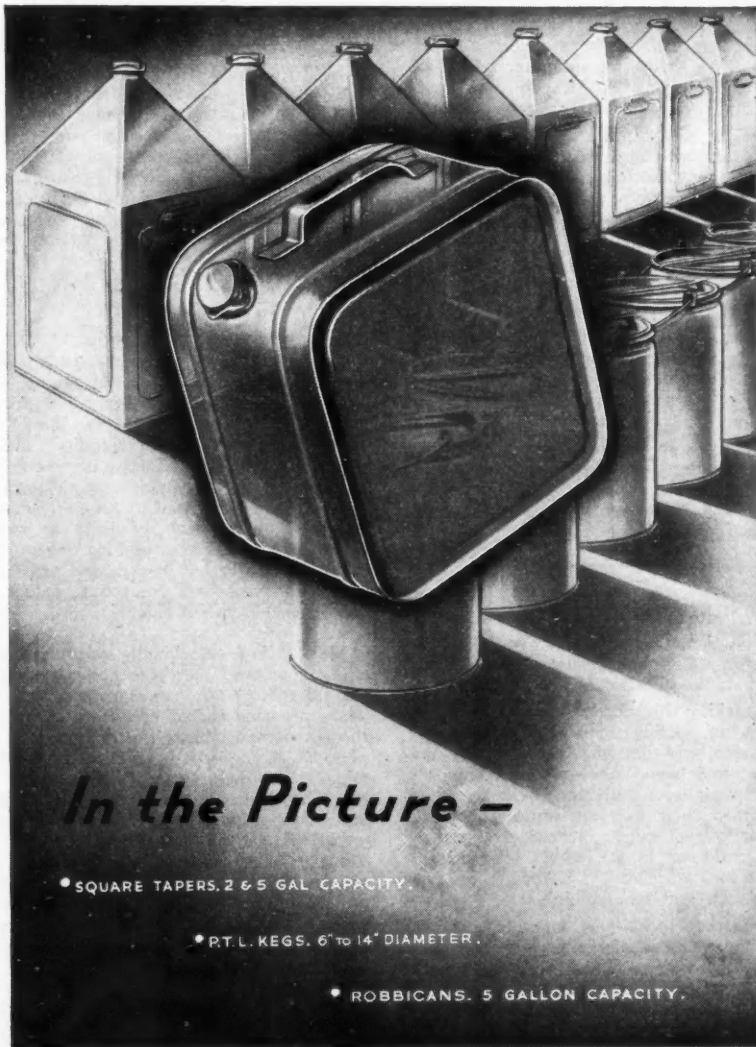
Estimates of the world production of tin in the first half of this year are stated to be 80,000 tons, an annual rate of 160,000 tons, as compared with 164,500 tons in 1951. Malayan output for the six months is estimated at 28,300 tons, total for the five months being 160 tons above the comparable period of 1951. Despite the lack of new prospecting due to the terrorist campaign 1952 production is unlikely to be affected, and a yield of 57,000 tons, only some 200 tons less than 1951, is expected. A world surplus of some 30,000 tons is anticipated for 1952, but most of this is likely to be absorbed into U.S. reserve stocks.

Boron & Lithium Research

Construction of a new research laboratory by the American Potash and Chemical Corporation was scheduled to begin in July. Site of the new laboratory, which will cost about \$300,000, will be at Whittier, California, and completion is expected early in 1953. The initial programme gives preference to production of boron and lithium compounds from the company's own raw materials. This will form part of a widened scheme of research on certain organic and inorganic chemicals for industry and agriculture in the western market.

Methane in Italy


The first national exhibition of methane will be held in Italy at Piacenza from 13-21 September. Latest developments in the research, transport and application of this natural gas will be displayed and emphasis will be laid on the importance of methane to Italian industry. Concurrently with the exhibition there will be a technical and economic conference to discuss problems, equipment and research.



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Fertiliser Prices in Europe

PRICES received by farmers throughout Europe for their major products are surveyed in a report on agricultural products and fertilisers in 1951-52, published on 11 August.

Dealing with fertilisers, the report which was made by the Agricultural Division of the Economic Commission for Europe states that between 1950-51 and 1951-52, prices increased considerably.

Changes were not all of the same order for all three plant nutrients. Potash was reduced in Denmark, Sweden and Italy, but all other prices were higher.

In most countries phosphatic fertilisers were increased much more than the other two types, and where nitrogen or potash prices were greater, it was only by a small margin.

The favourable supply situation in France resulted, however, in a rise in prices of phosphatic fertiliser of only 24 per cent compared with 36 per cent and 47 per cent for nitrogen and potash.

Monsanto Appointment

THE appointment of MR. J. W. URBAN as Director of Overseas Relations is announced by Monsanto Chemicals, Ltd.

In making this announcement MR. P. A. SINGLETON, acting managing director of Monsanto, said that while the company had always maintained a strong and vigorous export activity, it had been decided now to strengthen their overseas organisation still further to meet today's increasingly competitive conditions in the whole international sphere and especially in Europe, the Commonwealth and the Dominions.

Mr. Urban in his new duties will be responsible for personal contact with the company's agents in all countries, with Monsanto associated companies and with other important industrial concerns in overseas markets where their interests require such personal handling at executive level.

He will be located at the head office of the company at 8 Waterloo Place, London, S.W.1, and will move his office to that address at an early date.

Mr. Urban, who prior to taking up his

new appointment was a sales controller of Monsanto, joined the company in 1929 after having been associated with the chemical industry since 1919. For many years he has travelled extensively on the company's business and brings to his new appointment a very wide first-hand experience of foreign trade and affairs. During the war years he was attached to the Political Intelligence Department of the Foreign Office and served in the Middle East, Italy and Austria. He is a member of the Institute of Export, and a member of the Incorporated Sales Managers' Association.

Market Reports

LONDON.—The industrial chemicals market has been without any special feature this week, but the quiet conditions which prevail in most sections are not unusual during the holiday season. Overseas inquiry remains fairly good and a more active home demand is expected in the autumn. Calcium carbide has been increased to £30-£32 per ton; otherwise prices remain steady at recent levels. There has been nothing to report in the coal tar products market, and activity is still concerned with contract deliveries. Export trade remains quiet.

MANCHESTER.—Seasonally quiet conditions still obtain on the Manchester chemical market, though a fair movement of supplies of caustic soda and other alkalis, as well as the potash and ammonia compounds, is reported. Most prices have been fully maintained, the principal exceptions being the 35 per cent peroxide of hydrogen, which has been reduced by £3 to £153 per ton, and the oxides of zinc and other zinc compounds. There has been no more than a modest improvement in the demand for the general run of fertilisers. Among the by-products, crude and refined tar, creosote oil, and the phenols continue to find a reasonably good outlet.

GLASGOW.—Due to the annual holidays in the major consuming areas, business during the last two or three weeks has been quiet, but not unduly so. Interest should awaken again next week, but in view of the downward trend in prices and lack of orders in some industries, it is felt that trade on the whole will remain on the quiet side for some time to come. There is very little change in the export market.

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Publications & Announcements

THREE new products for laboratory use are announced by H. J. Elliott, Ltd., which is constantly working on new developments to solve problems of users of its apparatus.



The E-MIL plastic stopper, which has been used for four years in all E-MIL stoppered glassware, has now been extended to reagent bottles. The new plastic stopper, unlike glass, is not affected by certain chemicals and never 'freezes', 'leaks', or 'falls out.' The illustration shows the easy grip design which assists in its quick removal.

Other new items are separating funnels which are being fitted with the new E-MIL stopcock, the main feature of which is the key with the easy grip handle and spring-loaded retaining device, and the new E-MIL automatic pipette washer. The latter operates on a syphoning system, so that the water-level rises and falls in the container. Pipettes to be cleaned, are thus flushed continuously. The washer is manufactured in borosilicate glass so that boiling water can be safely used. The device is already available from stock, 'in any length.'

THE Simmerstat with its tap-like control has undoubtedly done more to popularise electric cooking than any other single development. Used in conjunction with a radiant plate its potentialities are realised to the full. Sunvic Controls Ltd., have therefore, introduced a range of one-circuit radiant boiling plates, and these are described and illustrated in their publication BP 10 (a). They have been designed for speed, and their low thermal capacity ensures minimum heating time when switched on and instant response to Simmerstat control. It is claimed that there is no wastage of electricity and the combination gives the most flexible control possible with the lowest current consumption. Full details can be obtained from the company, 10 Essex Street, London, W.C.2.

HAZARDS from the breakage of glass tubing are claimed to be eliminated by a tubing manipulator for protecting the hands described in the 'Safety Forum' of *Chemical and Engineering News* (30, 3041). Made of neoprene by Labline Inc., the Labline Grip-Safe provides a gentle but firm gripping action which permits sliding the tube into stoppers without the excessive or uneven pressure which breaks the tubing. Even if the tubing should break the manipulator protects the hands, wrists and knuckles from injury. It accommodates most sizes of commonly used tubing as well as irregular shaped tubing such as T's and L's, and may also be safely used to break tubing after marking with file or cutter.

WATER pollution abstracts are published monthly by the Water Pollution Research Staff of the Department of Scientific and Industrial Research. The latest list, February, 1952 (Vol. 25, No. 2), covers numbers 143-313 (HMSO, 3s. 6d.).

PROCESSING of sulphur due to its individual characteristics and the reactions which may be created by even careful handling, has always raised problems. As a result of considerable development work, a new approach to these problems has been made possible by the British 'Rema' Manufacturing Co., Ltd., and plants of new design are now in commercial operation with gratifying results. In an article, 'The Grinding and Air-Separation of Sulphur,' in *Edgar Allen News* for August (Vol. 31, No. 362), J. Davis, M.I.Chem.E., M.Inst.F., describe some of the 'Rema' processes and plant and shows how the tendencies for fire and explosion have been minimised.

FATTY ACIDS produced at its fractionating plant at Littleborough, Lancashire, are listed in a new brochure obtainable from Hess Products, Ltd., Leeds. The list at present covers 11 products under the heading saturated acids, four unsaturated, and one each under the headings of mixed fatty acids and miscellaneous. The brochure is of convenient size and loose-leaf form so that as new products become commercially available sheets can be added to keep the information up to date.



Illustration : Acid storage, supported by acid metering house and fume scrubbing tower at a South Wales steelworks.

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Expensive capital plant needs the assurance of long life and trouble-free service . . . yet how often does the problem of corrosion receive little more than cursory attention. Nutralines—which provide the complete answer to most corrosion problems—are a group of chemically-inert materials and processes used for the design and construction of acid-proof plant and equipment.

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Law & Company News

The following are taken from the printed reports, but we cannot be responsible for errors that may occur

Mortgages and Charges

(Note.—The Companies Consolidation Act of 1908 provides that every Mortgage or Charge, as described herein, shall be registered within 21 days after its creation, otherwise it shall be void against the liquidator and any creditor. The Act also provides that every company shall, in making its Annual Summary, specify the total amount of debt due from the company in respect of all Mortgages or Charges. The following Mortgages or Charges have been so registered. In each case the total debt, as specified in the last available Annual Summary, is also given—marked with an *—followed by the date of the Summary but such total may have been reduced.)

INDUSTRIAL CHEMICALS, LTD., London, W.C. (M., 16/8/52). 9 July, series of £5,000 debentures, present issue £2,250; general charge. *£11,250 debentures. 20 February, 1952.

Increases of Capital

The following increases of capital have been announced:—**J. K. INNES CO., LTD.**, from £6,000 to £15,000; **STYRENE CO-POLYMERS, LTD.**, from £250,000 to £350,000; **WITCO CHEMICAL CO., LTD.**, from £30,000 to £70,000; **BURROUGHS WELLCOME & CO. (AUSTRALIA), LTD.**, from £30,000 to £125,000.

New Registrations

Bignell Chemicals, Ltd.

Private company. (510,232). Capital £1,000. Manufacturers of chemicals, drugs, etc. Directors: J. B. Bignell and Mrs. M. Bignell. Reg. office: 6 Fitzroy Square, W.1.

Industrial Resins, Ltd.

Private company. Capital £100. Industrial, manufacturing, research, dispensing and analytical chemists. First directors are not named. Reg. Office: Argyle House, 29-31, Euston Road, N.W.1.

Kenworthy and Co. (Rochdale), Ltd.

Private company. (510,245). Capital £20,000. Analytical and hydrological chemists and consultants, etc. Directors: E. Kenworthy and Mrs. D. E. Kenworthy, W. F. Hemingway, M. Lindsay and J. Lindsay. Reg. office: Rugby Road, Rochdale.

Change of Name

Spear Laboratories, Ltd., of 37 Market Street, Paddock, Huddersfield, changed its name to **G. Wilson Marsden Ltd.**, on 18 July, 1952.

Company News

Oldham and Son, Ltd.

Reference to the rôle of the chemist, physicist and engineer in helping Britain to meet competition and sell her goods, services and techniques throughout the world, is made by Mr. John Oldham, O.B.E., chairman, in his statement to be presented at the 33rd annual general meeting of Oldham and Sons, Ltd., to be held at Denton, Manchester, on 29 August. The company's research programme has expanded continuously during the past five years, and an example of its long term research is the new 'Fibrak' battery separator now being produced both in the U.K. and the U.S.A. Overseas interests in France and South Africa were doing well and a new company, Oldham and Son (India), Ltd., had been got under way during the year. Net profit of the group (before taxation) for the year ended 31 March, 1952, was a record one of £319,706. It is proposed to increase the final dividend on ordinary stock to 20 per cent making 35 per cent for the year. An extraordinary meeting will be held at the conclusion of the general meeting to consider that £200,000 of the company's reserves be capitalised and applied in making a bonus issue of 4,000,000 ordinary shares of 1s. each in the proportion of one new share for each ordinary share held on 16 August, 1952.

International Nickel Company of Canada, Ltd.

The interim report of The International Nickel Company of Canada, Limited, and subsidiaries for the six months ended 30th June, 1952, issued by Dr. John F. Thompson, chairman of the board of directors, shows net earnings in terms of U.S. currency of \$33,538,227 after all charges, depreciation, depletion, taxes, and so on, equivalent, after preferred dividends, to \$2.23 per share on the common stock. For the corresponding period in 1951 the net earnings were \$29,385,046, or \$1.94 per share of common. In the three months ended 30 June, 1952, net earnings were \$17,191,909, equal to \$1.15 a share on the common, compared with \$16,346,318 or \$1.08 per share of common, in the first quarter of 1952 and \$14,653,656 or 97c. per share of common, in the second quarter* of 1951.

Fish Meal Bread

The South African Government is planning to introduce fish meal into the nation's bread. The natives do not get enough protein, vitamins, minerals and fats in their diet and their health suffers, so the South African Government has arranged to market brown bread enriched with peanut meal, powdered skimmed milk, buttermilk powder and calcium. Later the intention is to introduce a small proportion of a highly purified and palatable white fish meal flour which is said to be odourless and tasteless.

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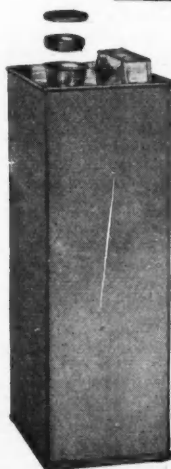
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2nd October, 1952.

SITUATIONS VACANT

The engagement of persons answering these advertisements must be made through a Local Office of the Ministry of Labour or a Scheduled Employment Agency if the applicant is a man aged 18-64 inclusive, or a woman aged 18-59 inclusive, unless he or she, or the employment, is exempted from the provisions of the Notifications of Vacancies Order, 1952.

BRITISH TAR PRODUCTS LIMITED, CADISHEAD, NR. MANCHESTER, require an **ASSISTANT CHIEF CHEMIST**, aged about 30-35, with University Degree or equivalent. Previous experience in the industry desirable. The post is permanent (with Pension Scheme) and involves control of the Works Laboratory, initially under the Chief Chemist, with prospect of early advancement. Opportunities for development and research.

CHEMICAL ENGINEER required for development work in connection with tar and benzole products, fully responsible for conducting research, including supervision of staff and designing plant and equipment. Very great scope for keen man, including operation of existing new plant and investigation into new products up to production stage. Permanent post (with pension scheme). Apply to **BRITISH TAR PRODUCTS, LTD., HAYES ROAD, CADISHEAD, MANCHESTER.**

EXPERIMENTAL OFFICERS AND ASSISTANT EXPERIMENTAL OFFICERS in various Government Departments. The Civil Service Commissioners invite applications for permanent appointments to be filled by competitive interview during 1952. A closing date for the receipt of applications earlier than December, 1952, may eventually be announced either for the competition as a whole or in one or more subjects.

The posts are divided between following main groups and subjects (a) Mathematical and Physical Sciences, (b) Chemistry and Metallurgy, (c) Biological Sciences, (d) Engineering subjects and (e) Miscellaneous (including e.g. Geology, Library and Technical Information Services).

Age Limits: For Experimental Officers, at least 26 and under 31 on 31st December, 1952; for Assistant Experimental Officers at least 18 and under 28 on 31st December, 1952. Extension for regular service in H.M. Forces.

Candidates must have obtained, or be taking examinations during 1952 with a view to obtaining, the Higher School Certificate with mathematics or a science subject as a principal subject, or the General Certificate of Education in appropriate subjects, or the Higher National Certificate or other specified qualifications. Candidates without such qualifications may be admitted exceptionally on evidence of suitable experience. Candidates over 20 will generally be expected to have higher qualifications.

Inclusive London salary scales:—

Experimental Officer £628-£786 (men); £533-£655 (women).

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Starting pay according to age up to 26. At 18, £275; at 26, £498 (men), £467 (women). Somewhat lower rates in the provinces.

Further particulars and application forms from the **CIVIL SERVICE COMMISSION, SCIENTIFIC BRANCH, TRINIDAD HOUSE, OLD BURLINGTON STREET, LONDON, W.1**, quoting No. 894-95/52. Completed application forms should be returned as soon as possible. 16327/100/JW.

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APV, Australia, and its associated company, Bryant Brothers of Sydney, require the services of an **ENGINEER** prepared to undertake sales campaigns in Australia for chemical plant and machinery. Applicants should be between 25-35 unmarried, and prepared to travel extensively. A sound knowledge of Engineering as applied to chemical plant, as well as sales drive and initiative, are essential qualifications. First-class prospects exist for any men capable of tackling successfully the problems associated with breaking into new technical markets. Apply to the **CHEMICAL ENGINEERING DEPARTMENT, THE A.P.V. COMPANY, LTD., WANDSWORTH PARK, S.W.18**, marking the envelope "AUSTRALIA."

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VARIOUS MIXERS FOR SALE

TWO 1½ size Harrison Carter **DISINTEGRATORS**.

One **FILTER PRESS** by Johnson, with 15 steam jacketed plates 13" x 13" and 16 frames.

One plate and frame **PRESS** by Johnson with 22 C.I. plates and 23 frames, 4-port type.

One Jeffrey **GRINDER**, 72 loose hammers, shaker feed

Two Turner 2½-sheet No. 2 **DRESSING MACHINES**, ball bearing

Size No. 3 Junior Hammamac **HAMMER MILL** with fan and cyclone, also No. 1 size Miracle **GRINDING MILLS**.

Robinson 3-sheet No. 1 size **CENTRIFUGAL DRESSING MACHINE** for dry powders, etc.

Two **ROTARY BOWL MIXERS**, 5 ft. diam., cast-iron built, inclined agitators by Baker Perkins

One excellent **EVAPORATING UNIT**, comprising Copper Vessel, 4 ft. diam. by 5 ft. 6 in. deep, jacketed on the bottom, with copper swan-neck, C.I. catch-pot, vacuum pump and fittings including thermometer and gauge.

Large unjacketed **WERNER MIXER**, belt and gear driven, hand tipping, double "Z" arms, pans 31 in. by 14 in. by 36 in. deep.

No. 200 One nearly new **WERNER PFLEIDERER JACKETED MIXER OR INCORPORATOR**. Low type, with C.I. built mixing chamber. 28 in. by 29 in. by 27 in. deep, with double "U"-shaped bottom which is jacketed, and double fish-tail or fin-type agitators geared together at one side, with belt-driven friction pulleys, 34 in. diam. by 5 in. face, with hand-wheel operation and hand-operated screw tilting gear. Machine fitted with machine-cut gears, covers, gear guard, cast-iron baseplate, and measuring overall approximately 7 ft. by 6 ft. by 4 ft. high to the top of the tipping screw

No. 209 One **HORIZONTAL "U"-SHAPED MIXER**, steel built, riveted, measuring about 8 ft. 3 in. long by 3 ft. wide by 3 ft. 3 in. deep, with horizontal shaft, fitted with bolted-on mixing arms about 18 in. long by 4 in. wide, with intermediate breakers, and driven at one end by a pair of spur gears, with countershaft, fast and loose belt pulleys; outer bearing and plug cock type outlet at the opposite end, mounted on two cradles fitted to two R.S.J. running from end to end.

One **FILTER PRESS**, fitted 68 wood recessed plates, 2 ft. 8 in. square, centre fed, with enclosed bottom corner delivery, cloth clips and belongings.

One **DEHNE FILTER PRESS**, cast-iron built, fitted 45 recessed ribbed plates, 2 ft. 8 in. by 2 ft. 8 in. by 1½ in., with bottom corner feed, cloth clips and bottom corner separate outlets, angle lever closing gear, etc.

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Further details and prices upon application.

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FIVE new **FURNACE RETORTS**, 8 ft. diam., 6 ft. 8 in. deep, approx. 8 tons each.

BENZOLE WASHER, lead lined, agitating gear, 7 ft. diam., 6 ft. 6 in. deep.

FIVE Dish-ended **NAPHTHA TANKS**, 18 ft. 6 in. long by 4 ft. 4 in. diam., two having agitators.

NINE New Welded **TANKS**, 13 ft. 6 in. long, 7 ft. diam., 3,100 gallons each.

TWO 35 ft. long by 9 ft. diam. Lead-lined **TANKS**, Stainless Steel **FILTER TANK**, 3 ft. 6 in. diam.

TWO Stainless **CONICAL HOPPERS**, 1,200 gallons, 8 ft. 3 in. diam., overall depth, 7 ft. 6 in.

TWO Broadband **WATER-DRIVEN CENTRIFUGES**, 30 in. diam., 12 in. deep, 1,150 r.p.m., 150 lb. pressure.

FOUR Papier-mache **O.T. TANKS**, 8 ft. 6 in. diam., 8 ft. deep. (Unused.)

SIX **O.T. TANKS**, 7 ft. diam. 14ft. deep, lined inside with acid-resisting bricks.

SIX Aluminium **CONDENSERS**, 14 ft. long by 2 ft. 6 in. diam. 386 Tubes, ½ in. o.d.

FOUR Rectangular Lead-lined **TANKS**, 8 ft. by 4 ft. 6 in. by 2 ft. 6 in.

FORTY Riveted **RECEIVERS**, 8 ft. 6 in. long, 5 ft. 6 in. diam., 75 lbs. w.p.

CAST-IRON PIPES AND FITTINGS, 200 tons.

VALVES in Stainless, Gunmetal, Enamel Lined.

Free Catalogue, "Watkins Machinery Record," available.

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DELAFLA, THE INERT FILLER. Used in the manufacture of Fertilisers, Insecticides, Paints, Plastics and Insulating and Sealing Compounds. Prompt supplies in a wide range of fineness grades. **THE DELABOLE SLATE CO., LTD. DELABOLE, CORNWALL.**

GRAVITY Roller Conveyor several lengths, Rolls. 2½ in. diam. by 16 in. 3 in. centres. Good condition. **THOMPSON & SON (MILLWALL), LIMITED, CUBA STREET MILLWALL E.14. (Tel. East 1844.)**

SCREENLESS PULVERIZERS for fine grinding of Chemicals. Also **CYCLONES, ROTARY VALVE FEEDERS**. Callow (Engrs.) Ltd. Kirby Trading Est., Liverpool.

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200 R.S. Cylindrical Storage **TANKS** up to 12,000 gallons.

Unused 4,000 gal. **W.S. TANKS**, 15 ft. by 7 ft. ½ in. plate.

Five All-Aluminium **CONDENSERS**, 130 and 245 sq. ft. Stainless Steel Gas-heated Jacketed **MIXER**, 22½ in. c., 36 in. deep.

Jacketed Cylindrical Vacuum **PAN**, 7 ft. by 2 ft. 6 in., with pump and condenser.

DISINTEGRATORS, 24 in., 22 in., 18 in. and 12 in. diam. "Werner" and Twin "Z" **MIXER** (jacketed and plain) up to 10 cwt. capacity.

Stainless Steel Revolving **DRUM**, 4 ft. diam. by 2 ft. **PUMPS, BOILERS, HYDROS, STEAM AND DIESEL ENGINES**, etc.

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1 Barron "D" **MIXER, TROUGH** 30 in. by 18 in. by 18 in. Vee-belt drive to 2 H.P. motor, 750 revs, 400/3/50. As new.

One Werner Type **MIXER, TROUGH** 36 in. by 30 in. by 24 in. Twin "Z"-blades, power tilted, fast and loose pulley drive.

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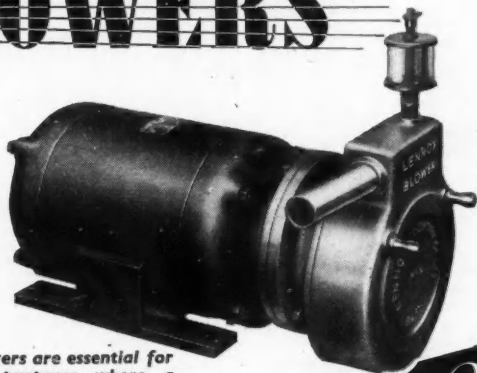
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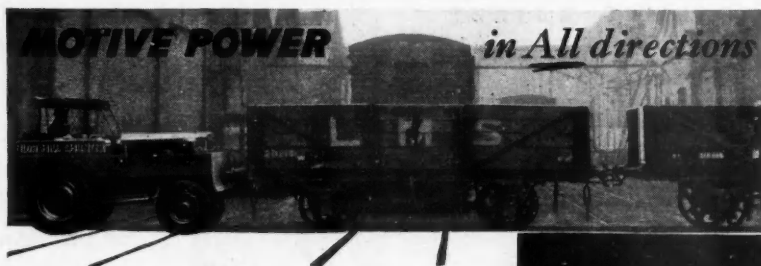
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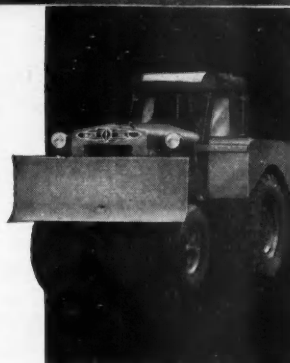


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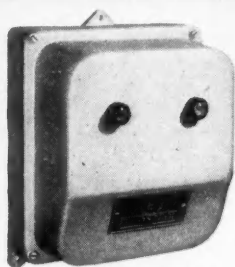
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